Agilent 1200 Series Refractive Index Detector

User Manual

Agilent Technologies

Notices

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Manual Structure

The **Service Manual G1362-90110** (English) contains the complete information about the Agilent 1200 Series Refractive Index Detector. It is available as Adobe Reader file (PDF) only.

The **User Manual G1362-90010** (English) and its localized versions contain a subset of the Service Manual and is shipped with the detector in printed matter.

Latest versions of the manuals can be obtained from the Agilent web.

Software Revision

This guide is valid for B.01.xx revisions of the Agilent 1200 Series ChemStation Software, where xx refers to minor revisions of the software that do not affect the technical accuracy of this guide.

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A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

In This Guide...

This manual contains user information about the Agilent 1200 Series Refractive Index Detector. The manual describes the following:

1 Introduction to the Refractive Index Detector

This chapter gives an introduction to the Refractive Index Detector.

2 Site Requirements and Specifications

This chapter provides information on environmental requirements, physical and performance specifications.

3 Installing the Refractive Index Detector

This chapter provides information on unpacking, checking on completeness, stack considerations and installation of the detector.

4 Using the Refractive Index Detector

This chapter provides information on how to set up the detector for an analysis and explains the basic settings.

5 Optimizing the Refractive Index Detector

This chapter provides information on how to optimize the detector.

6 Troubleshooting Overview

This chapter gives an overview about the troubleshooting and diagnostic features.

7 Repairing the Refractive Index Detector

This chapter provides general information on repairing the detector.

8 Maintaining the Refractive Index Detector

This chapter describes the maintenance of the detector.

A Safety Information

This chapter provides additional information on safety, legal and web.

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This chapter gives an introduction to the Refractive Index Detector.

Introduction to the Refractive Index Detector

The detector is designed for highest optical performance, GLP compliance and easy maintenance. It includes the following features:

- advanced temperature controlled detector optics ready to use within two hours of installation
- automatic zero and automatic purge combined with a recycle valve for automatic solvent recycling allow uninterrupted operation
- durable tungsten lamp with a life expectancy of 40,000 hours
- automatic light intensity control circuit to ensure the optimum performance of the optics
- integrated diagnostics for efficient troubleshooting
- built-in refractive index calibration
- front access to valves and capillaries for easy maintenance

For specifications, see “Performance Specifications” on page 29.

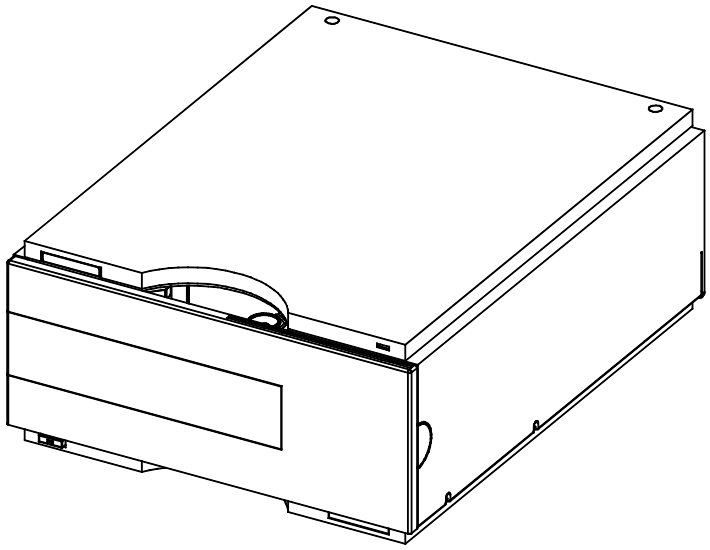


Figure 1 The Agilent 1200 Series Refractive Index Detector

How the Detector Operates

Refractive Index Detection

Refractive index

When a beam of light passes from one medium into another, the wave velocity and direction changes. The change in direction is called refraction. The relationship between the angle of incidence and the angle of refraction is expressed in Snell's Law of refraction.

Snells's Law

$$n = \frac{n_2}{n_1} = \frac{\sin\alpha_1}{\sin\alpha_2}$$

Where:

n = Refractive index of medium 1 relative to medium 2

n_2 = Refractive index of medium 2

n_1 = Refractive index of medium 1

α_1 = angle of incident light in medium 1

α_2 =angle of refraction in medium 2

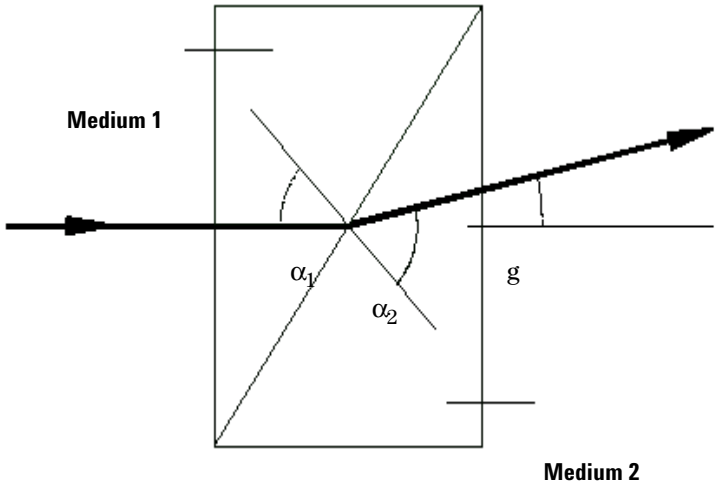


Figure 2 Light Refraction

According to the formula below small angles of external deflection are proportional to the difference between the refractive indices of medium 1 and medium 2.

$$\tan\gamma = \frac{n_1 - n_2}{n_1}$$

Where:

γ = angle of external deflection

Factors that Affect Refractive Index

The refractive index of a medium is affected by a number of factors;

1 Wavelength

   The refractive index varies with changes in the wavelength of the incident light beam.

2 Density

As the density of the medium changes the refractive index changes. At a fixed wavelength of incident light the changes in refractive index are generally linear in relation to the changes in medium density.

The density of a medium will be affected by the following factors:

- Composition (if not a pure substance)
- Temperature
- Pressure

Detection Principle

Detector Design

The Agilent 1200 Series refractive index detector is a differential refractometer that measures the deflection of a light beam due to the difference in refractive index between the liquids in the sample and reference cells of a single flow cell.

A beam of light from the lamp passes through a flow cell which is separated diagonally into sample and reference cells. At the rear of the flow cell a mirror reflects the light back through the flow cell and via a zero glass, which affects the path of the light beam, to the light receiver. The light receiver has two diodes each of which produces an electrical current proportional to the amount of light that falls upon it (see Figure 3).

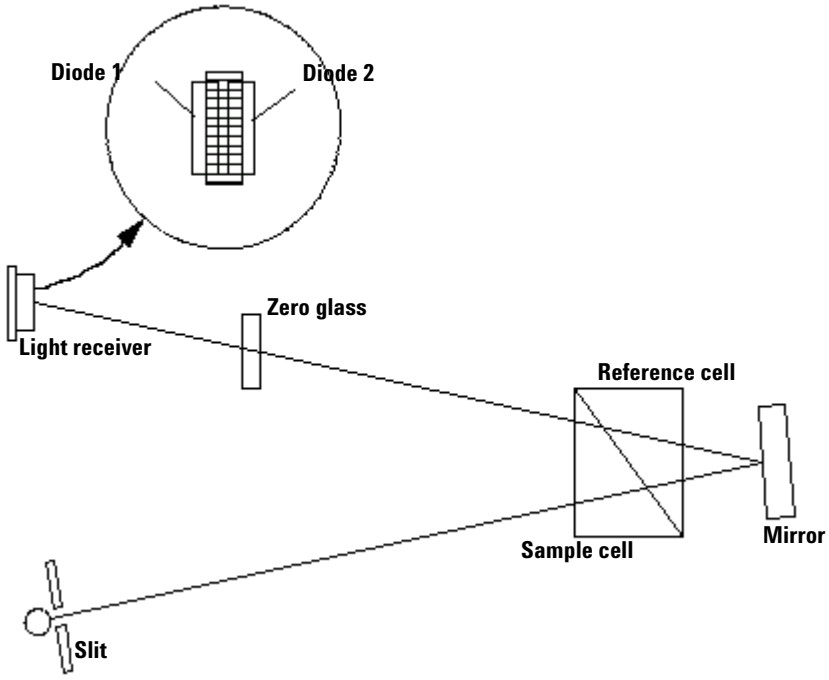


Figure 3 Detection Principle

Measurements

Initially both sample and reference cell are flushed with mobile phase. The reference cell is then closed and solvent flows only through the sample cell. The refractive index of the mobile phase in both cells is the same and the position of the zero glass can be adjusted so that the detector is in optical balance with an equal amount of light falls on each diode.

When sample elutes from the column into the sample cell the refractive index of the cell contents changes. The change in refractive index deflects the light beam as it passes through the flow cell resulting in an unequal amount of light falling on each diode. The change in current from the diodes that this causes is amplified and used to produce the calibrated detector signal. This signal expressed, as nano Refractive Index Units (nRIU), corresponds to the difference between the refractive index of sample in the sample cell and the mobile phase in the reference cell.

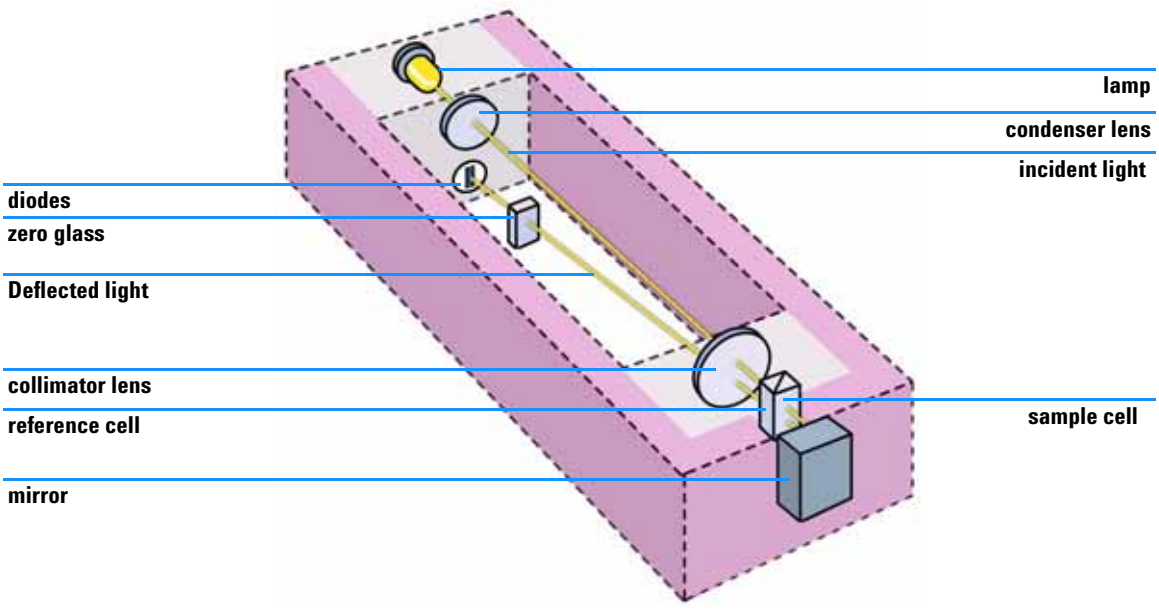


Figure 4 Optical Path

Flow Path

Flow Path

The column eluent enters the optical unit through the in port and passes through a heat exchanger. The combination of the heat exchanger and control of the optical unit temperature in the range of 5 °C above ambient to 55 °C minimizes changes in refractive index due to temperature variations. The eluent flows through the sample cell and via the same heat exchanger to the purge valve. With the purge valve in the OFF position the eluent passes to the recycle valve. If the recycle valve is also in the OFF/WASTE position the eluent will flow via the waste port into the waste container.

If the recycle valve is in the ON/BOTTLE position the eluent will flow via the recycle port back to the solvent bottle. The recycle valve can be manually set to the ON or OFF position or the **Automatic recycling after analysis** mode can be enabled. In this mode the recycle valve will automatically switch to the ON position after each analysis has been completed and return to the OFF position before the next analysis starts. Using this mode provides the benefits of uninterrupted flow through the detector without the problems of excessive solvent usage or the contamination of mobile phase with recycled sample compounds.

If the purge valve is in the *on* position the eluent cannot pass immediately to the recycle valve but will instead flow via a second heat exchanger through the reference cell and then into the recycle valve (see Figure 5). Periodically switching the purge valve to the *on* position while only mobile phase is flowing will ensure that the liquid in the reference cell is as similar as possible to the flowing solvent. The purge valve can be manually set to the *on* position for a defined time or the **Automatic purge** mode can be enabled. In this mode the purge valve will automatically switch to the ON position for a defined **purgetime** prior to the start of each analysis. If a **purgetime** is set then a **waittime** must also be set to allow the detector baseline to stabilize after the switching of the purge valve position.

After both the purgetime and waittime have been completed the analysis will start. If the **Automatic zero before analysis** mode is enabled the detector output will be set to zero immediately before the analysis begins.

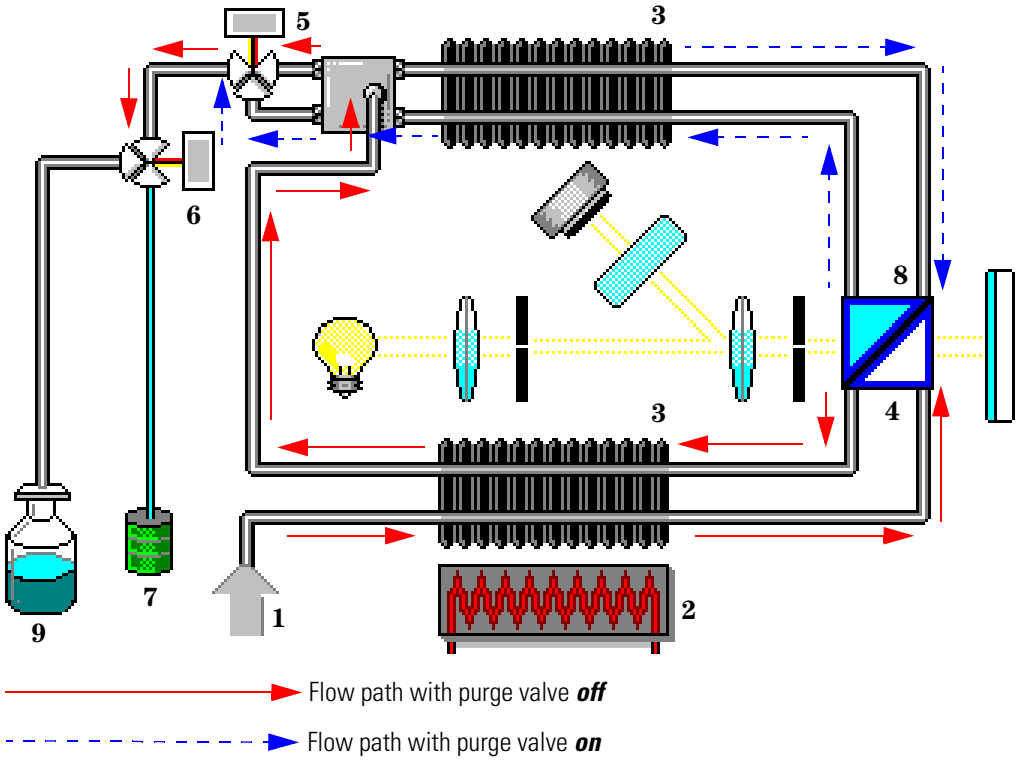
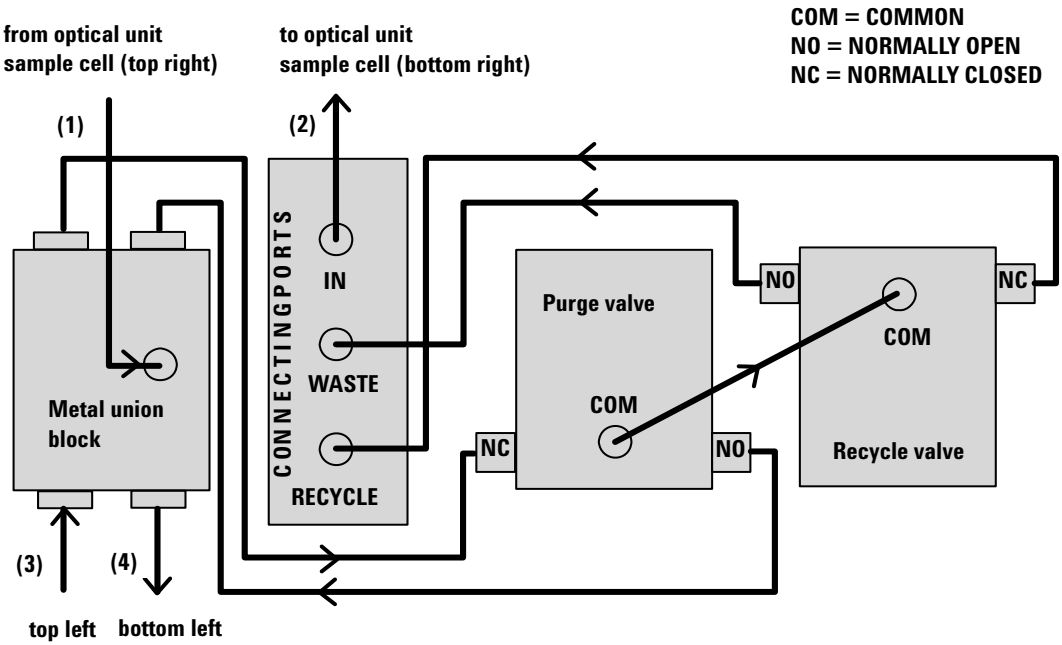


Figure 5 Flow Path

1 Flow in
2 Heater
3 Heat exchanger
4 Sample cell
5 Purge valve
6 Recycle valve
7 Waste container
8 Reference cell
9 Solvent bottle



optical unit (from and to reference cell)

Capillaries (1) to (4) are part of the optical unit assembly. They are made of SST with an ID of 1.0 mm, except for (2), which has an ID of 0.2 mm. All other tubings (to and from the purge and the reference valve) are made of Teflon and are available as a kit (PN G1362-68709)

Figure 6 G1362A Physical Plumbing Connections

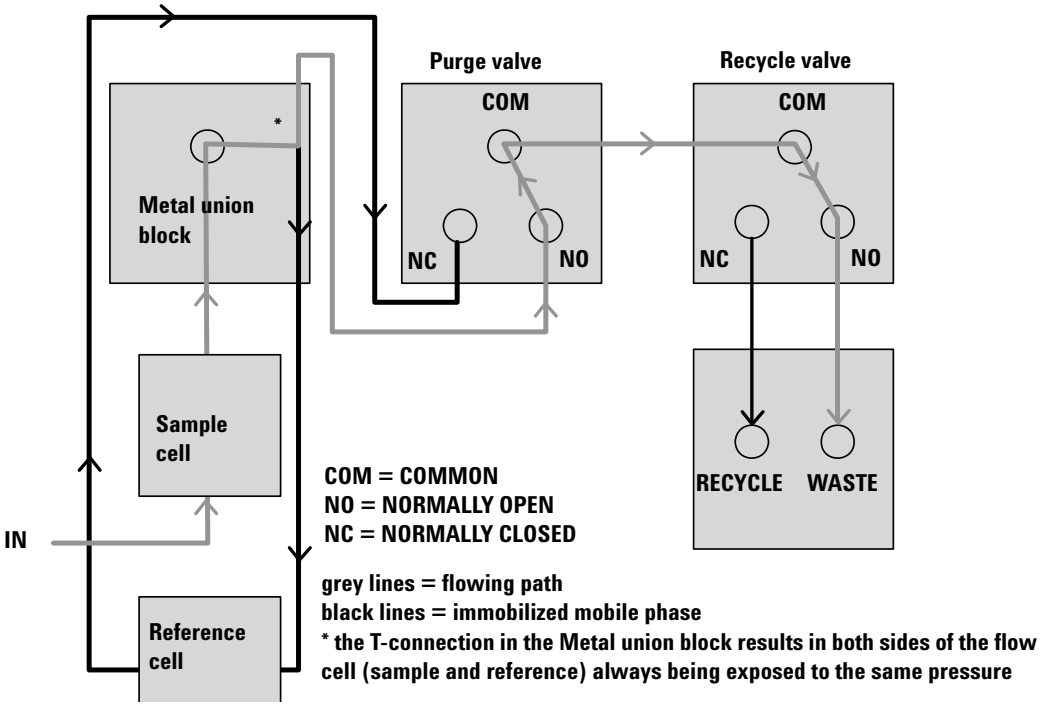


Figure 7 Flow path with the Purge- and Recycle-Valves = OFF

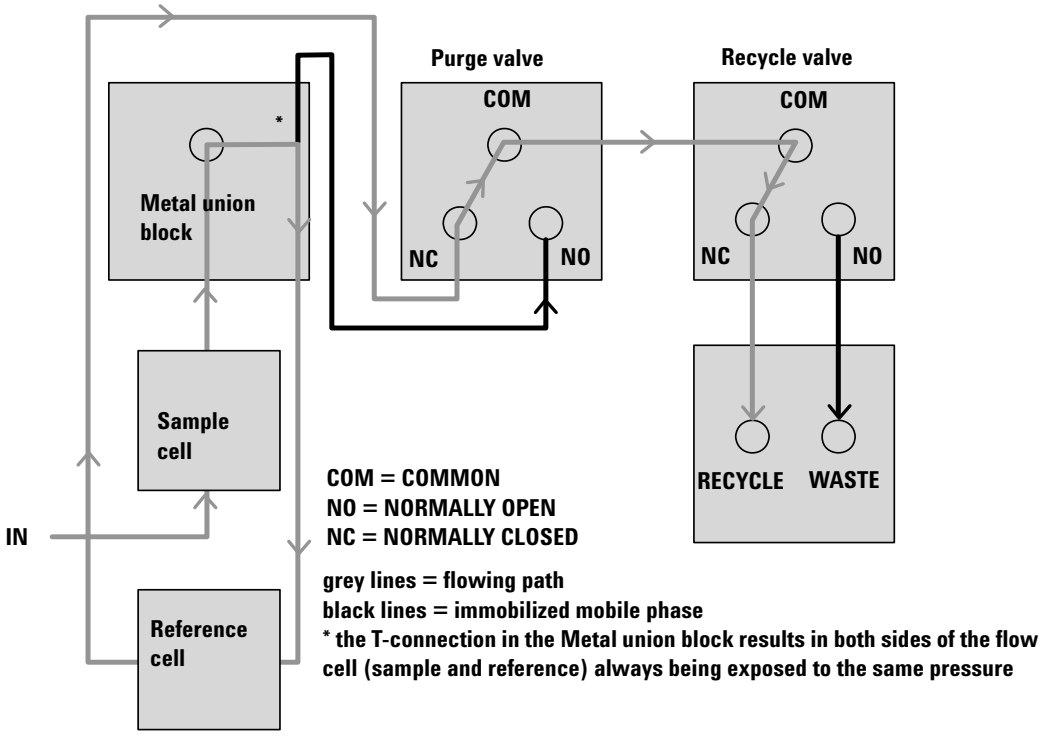


Figure 8 Flow path with the Purge- and Recycle-Valves = ON

Electrical Connections

- The GPIB connector is used to connect the detector to the with a computer. The address and control switch module next to the GPIB connector determines the GPIB address of your detector. The switches are preset to a default address (Table 24 in the Service Manual or "Setting the 8-bit Configuration Switch" in the Service Manual) and is recognized once after power is switched ON.
- The CAN bus is a serial bus with high speed data transfer. The two connectors for the CAN bus are used for internal Agilent 1200 Series module data transfer and synchronization.
- One analog output provides signals for integrators or data handling systems.
- The interface board slot is used for external contacts and BCD bottle number output or LAN connections.
- The REMOTE connector may be used in combination with other analytical instruments from Agilent Technologies if you want to use features such as start, stop, common shut down, prepare, and so on.
- With the appropriate software, the RS-232C connector may be used to control the module from a computer through a RS-232C connection. This connector is activated and can be configured with the configuration switch next to the GPIB connector (see "Communication Settings for RS-232C Communication" in the Service Manual). See your software documentation for further information.
- The power input socket accepts a line voltage of 100–120 or 220–240 volts AC ± 10% with a line frequency of 50 or 60 Hz. Maximum power consumption is 160 VA. There is no voltage selector on your module because the power supply has wide-ranging capability. There are no externally accessible fuses, because automatic electronic fuses are implemented in the power supply. The security lever at the power input socket prevents the module cover from being taken off when line power is still connected.

WARNING **Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.**

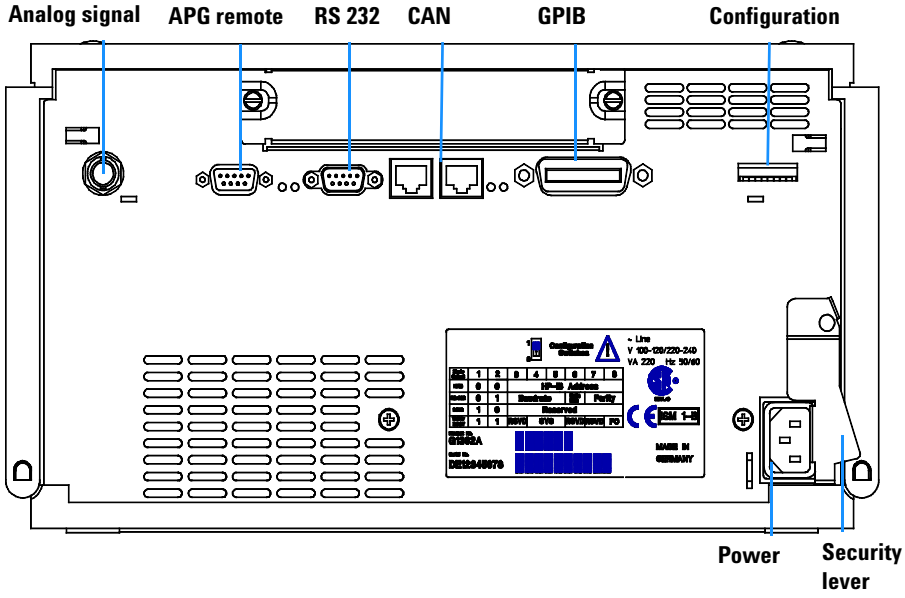


Figure 9 Electrical Connections

Instrument Layout

The industrial design of the module incorporates several innovative features. It uses Agilent's E-PAC concept for the packaging of electronics and mechanical assemblies. This concept is based upon the use of expanded polypropylene (EPP) layers foam plastic spacers in which the mechanical and electronic boards components of the module are placed. This pack is then housed in a metal inner cabinet which is enclosed by a plastic external cabinet. The advantages of this packaging technology are:

- virtual elimination of fixing screws, bolts or ties, reducing the number of components and increasing the speed of assembly/disassembly,
- the plastic layers have air channels molded into them so that cooling air can be guided exactly to the required locations,
- the plastic layers help cushion the electronic and mechanical parts from physical shock, and
- the metal inner cabinet shields the internal electronics from electromagnetic interference and also helps to reduce or eliminate radio frequency emissions from the instrument itself.

Early Maintenance Feedback (EMF)

Maintenance requires the exchange of components which are subject to wear or stress. Ideally, the frequency at which components are exchanged should be based on the intensity of usage of the detector and the analytical conditions, and not on a predefined time interval. The early maintenance feedback (EMF) feature monitors the usage of specific components in the instrument, and provides feedback when the user-selectable limits have been exceeded. The visual feedback in the user interface provides an indication that maintenance procedures should be scheduled.

EMF Counters

The detector provides one EMF counters for the reference liquid age. The counters increment with the time that liquid remains in the reference cell, and can be assigned a maximum limit which provides visual feedback in the user interface when the limit is exceeded. The counters are reset to zero after the reference cell is purged.

Using the EMF Counters

The user-selectable EMF limits for the EMF counters enable the early maintenance feedback to be adapted to specific user requirements. The useful counter time since last purge is dependent on the requirements for the analysis, therefore, the definition of the maximum limits need to be determined based on the specific operating conditions of the instrument.

Setting the EMF Limits

The setting of the EMF limits must be optimized over one or two maintenance cycles. Initially, no EMF limit should be set. When instrument performance indicates maintenance is necessary, take note of the values displayed by reference liquid age counters. Enter this values (or a value slightly less than

the displayed values) as an EMF limit, and then reset the EMF counter to zero. The next time the EMF counter exceed the new EMF limit, the EMF flag will be displayed, providing a reminder that maintenance needs to be scheduled.

1 Introduction to the Refractive Index Detector

2
Site Requirements and Specifications

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Performance Specifications 29

This chapter provides information on environmental requirements, physical and performance specifications.

Site Requirements

Power Consideration

The detector power supply has wide ranging capabilities and accepts any line voltage in the range mentioned in Table 1 on page 28. Consequently, there is no voltage selector in the rear of the detector. There are also no externally accessible fuses, because automatic electronic fuses are implemented in the power supply.

WARNING **To disconnect the detector from line, unplug the power cord. The power supply still uses some power, even if the power switch on the front panel is turned off.**

WARNING **Shock hazard or damage of your instrumentation can result, if the device is connected to a line voltage higher than specified.**

CAUTION Make sure to have easy access to the power cable of the instrument, in order to disconnect the instrument from line.

Power Cords

Different power cords are offered as options with the detector. The female end of all power cords is identical. It plugs into the power-input socket at the rear of the detector. The male end of each power cord is different and designed to match the wall socket of a particular country or region.

WARNING **Never operate your instrumentation from a power outlet that has no ground connection. Never use a power cord other than the Agilent Technologies power cord designed for your region.**

WARNING **Never use cables other than the ones supplied by Agilent Technologies to ensure proper functionality and compliance with safety or EMC regulations.**

Bench Space

The detector dimensions and weight (see Table 1 on page 28) allow you to place the detector on almost any desk or laboratory bench. It needs an additional 2.5 cm (1.0 inches) of space on either side and approximately 8 cm (3.1 inches) in the rear for air circulation and electric connections.

If the bench should carry an Agilent 1200 Series system, make sure that the bench is designed to bear the weight of all modules.

The detector should be operated in a horizontal position.

Environment

Your detector will work within the specifications at ambient temperatures and relative humidity described in Table 1 on page 28.

ASTM drift tests require a temperature change below 2 °C/hour (3.6 °F/hour) over one hour period. Our published drift specification (refer also to "Performance Specifications" on page 29) is based on these conditions. Larger ambient temperature changes will result in larger drift.

Better drift performance depends on better control of the temperature fluctuations. To realize the highest performance, minimize the frequency and the amplitude of the temperature changes to below 1 °C/hour (1.8 °F/hour). Turbulences around one minute or less can be ignored.

CAUTION Do not store, ship or use your detector under conditions where temperature fluctuations could cause condensation within the detector. Condensation will damage the system electronics. If your detector was shipped in cold weather, leave it in its box and allow it to warm up slowly to room temperature to avoid condensation.

NOTE The G1362A RID is designed to operate in a typical electromagnetic environment (EN61326-1) where RF transmitters, such as mobile phones, should not be used in close proximity.

Physical Specifications

Table 1 Physical Specifications Agilent 1200 Series Refractive Index Detector

Type	Specification	Comments
Weight	17 kg (38 lbs)	
Dimensions (width × depth × height)	345 × 435 × 180 mm (13.5 × 17 × 7 inches)	
Line voltage	100 – 240 VAC, ± 10 %	Wide-ranging capability
Line frequency	50 or 60 Hz ± 5 %	
Power consumption	160 VA	Maximum
Ambient operating temperature	0 – 55 °C (32 – 131 °F)	
Ambient non-operating temperature	-40 – 70 °C (-4 – 158 °F)	
Rel. Humidity	< 95%, at 25 – 40 °C (77 – 104 °F)	Non-condensing
Operating altitude	Up to 2000 m (6500 ft.)	
Non-operating altitude	Up to 4600 m (14950 ft.)	For storing the detector
Safety standards: IEC, CSA, UL, EN	Installation category II, pollution degree 2	For indoor use only

Performance Specifications

Table 2 Performance Specifications Agilent 1200 Series Refractive Index Detector

Type	Specification	Comments
Detection type	Refractive Index	
Refractive index range	1.00 - 1.75 RIU, calibrated	
Measurement range	+/- 600 x 10^{-6} RIU	
Optical zeroing		via set screw
Optics temperature control	5 ° C above ambient to 55 ° C	
Sample cell	volume 8uL maximum pressure 5 bar (0.5Mpa) maximum flow rate 5mL/minute	
Valves	Automatic purge and automatic solvent recycle	
Volumes	Inlet port to sample cell 62uL, inlet port to outlet port 590uL	
Liquid contact materials	316 stainless steel, teflon and quartz glass	
pH range	2.3 - 9.5	
Performance specifications	Short term noise < +/- 2.5 x 10^{-9} RIU Drift < 200 x 10^{-9} RIU/hour	see note below this table
Time programmable parameters	polarity, peak width	
Detector zero	automatic zero before analysis	

Table 2 Performance Specifications Agilent 1200 Series Refractive Index Detector (continued)

Type	Specification	Comments
Control and data evaluation	Parameter entry, signal display, on-line help and diagnostics with the Agilent 1200 Series Control Module. Optional PCMCIA card for method, sequence and logbook storage and transfer. Agilent ChemStation for LC PC based software for control and data evaluation.	
Analog outputs	Recorder/integrator: 100 mV or 1 V, output range selectable, one output	
Communications	Controller-area network (CAN), GPIB, RS-232C, LAN, APG Remote: ready, start, stop and shut-down signals	
Safety and maintenance	Extensive diagnostics, error detection and display (through control module and ChemStation), leak detection, safe leak handling, leak output signal for shutdown of pumping system. Low voltages in major maintenance areas.	
GLP features	Early maintenance feedback (EMF) for continuous tracking of instrument usage with user-selectable limits and feedback messages. Electronic records of maintenance and errors. Automated operational qualification/performance verification (OQ/PV).	
Housing	All materials recyclable.	

Table 2 Performance Specifications Agilent 1200 Series Refractive Index Detector (continued)

Type	Specification	Comments
Environment:	0 to 55 ° C constant temperature at <95% humidity (non-condensing)	
Dimensions:	180 mm x 345 mm x 435 mm (7 x 13.5 x 17 inches) (height x width x depth)	
Weight	17 kg (38 lbs)	

NOTE

Based on ASTM method E-1303-95 “Practice for Refractive Index Detectors used in Liquid Chromatography”.Reference conditions; optics temperature 35 ° C, response time 4 s, flow 1.0 mL/min LC-grade Water, restriction capillary, column compartment temperature 35 ° C, Agilent 1200 Series on-line vacuum degasser, pump and thermostatted column compartment. Instrument equilibrated for 2 hours.

3
Installing the Refractive Index Detector

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Installing the Detector 39
Flow Connections to the Detector 42

This chapter provides information on unpacking, checking on completeness, stack considerations and installation of the detector.

Unpacking the Detector

Damaged Packaging

If the delivery packaging shows signs of external damage, please call your Agilent Technologies sales and service office immediately. Inform your service representative that the detector may have been damaged during shipment.

CAUTION If there are signs of damage, please do not attempt to install the detector.

Delivery Checklist

Ensure all parts and materials have been delivered with the detector. The delivery checklist is shown below. Please report missing or damaged parts to your local Agilent Technologies sales and service office.

Table 3 Detector Checklist

Description	Quantity
Detector	1
Power cable	1
User Manual	1
Accessory kit (see Table 4)	1

Detector Accessory Kit Contents

Table 4 Accessory Kit Contents (Part Number G1362-68705)

Description	Part Number	Quantity
Interface tubing kit includes:	G1362-68706	1
• Ferrule 1/8″	0100-1700	2
• Nut 1/8″ PPS	0100-1708	2
• 2 m Tubing flexible	0890-1760	2
Interfacing capillary, 400mm lg, 0.17mm i.d. includes:	G1362-87300	1
• Hand tight fitting (reorder pack with 10 pcs.)	5062-8541	2
Restriction capillary, 3700mm lg, 0.17mm i.d. includes:	G1362-87301	1
• Hand tight fitting (reorder pack with 10 pcs.)	5062-8541	2
CAN cable	5181-1516	1
PEEK adapter to pump's active inlet valve	0100-1847	1

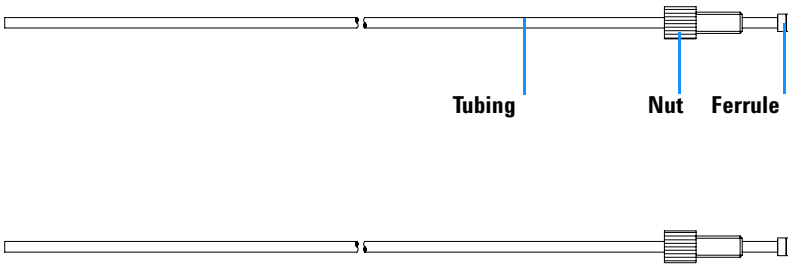


Figure 10 Interface tubing kit Parts

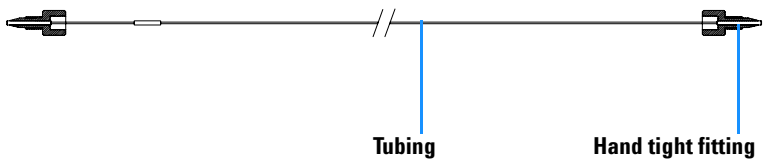


Figure 11 Interfacing Capillary Parts

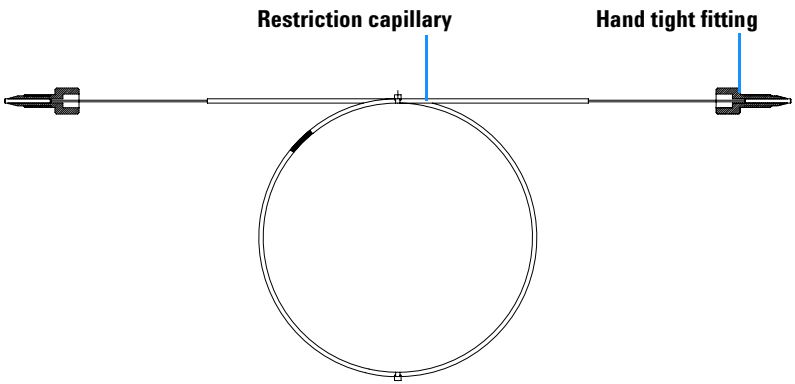


Figure 12 Restriction Capillary Parts

Optimizing the Stack Configuration

If your detector is part of a complete Agilent 1200 Series system, you can ensure optimum performance by installing the following configuration. This configuration optimizes the system flow path, ensuring minimum delay volume.

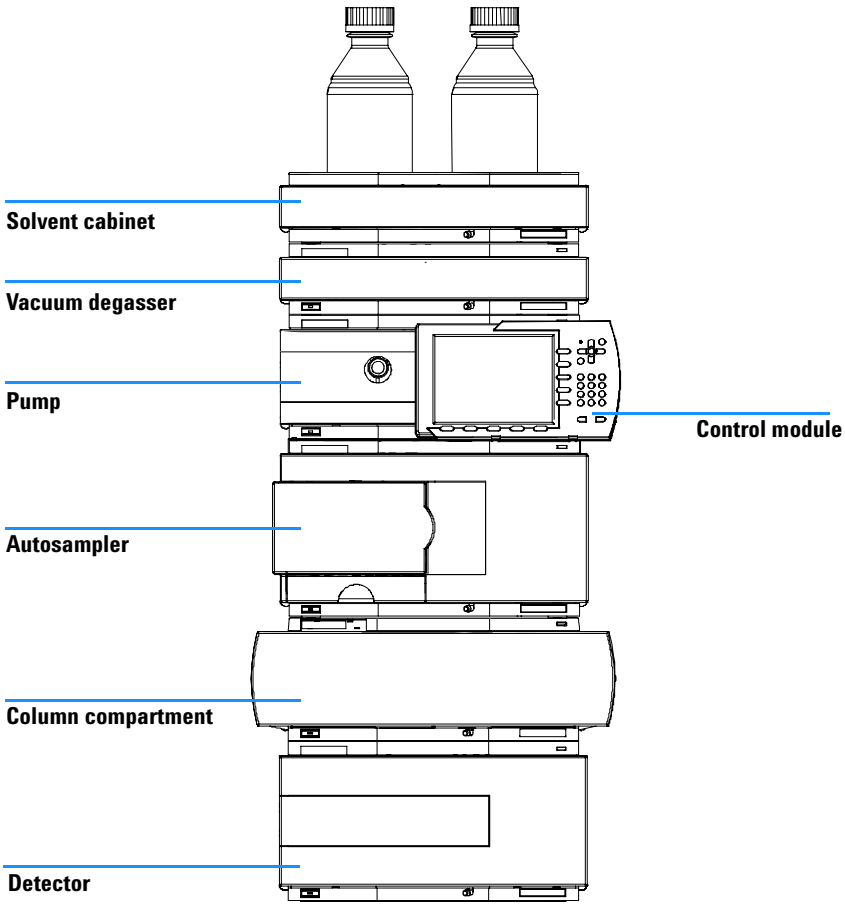


Figure 13 Recommended Stack Configuration (Front View)

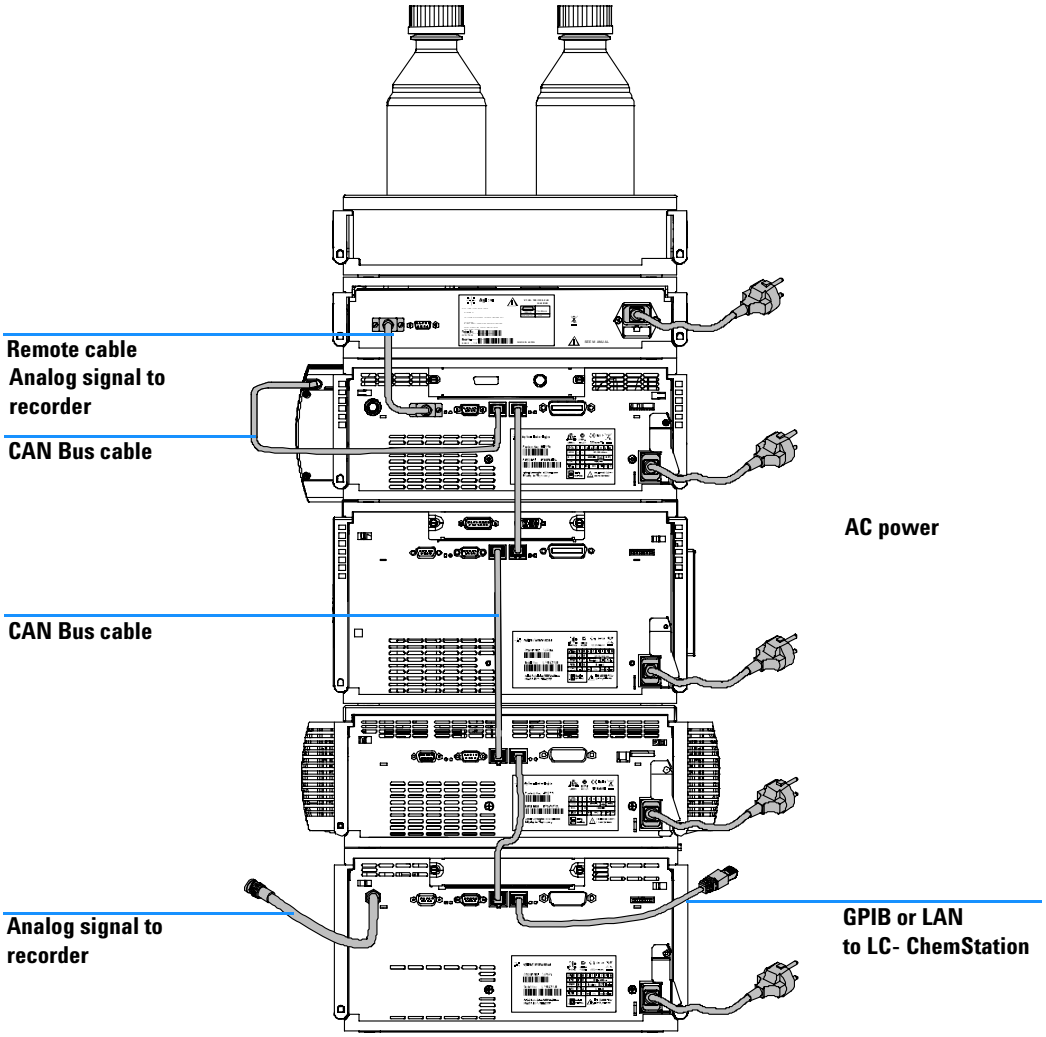


Figure 14 Recommended Stack Configuration (Rear View)

Installing the Detector

Preparations	Locate bench space Provide power connections Unpack the detector
Parts required	Detector Power cord, for other cables see below and "Cable Overview" in the Service Manual.

1 Install the LAN interface board in the detector (if required), see "Replacing the Interface Board" in the Service Manual.

2 Place the detector in the stack or on the bench in a horizontal position.

3 Ensure the line power switch at the front of the detector is OFF.

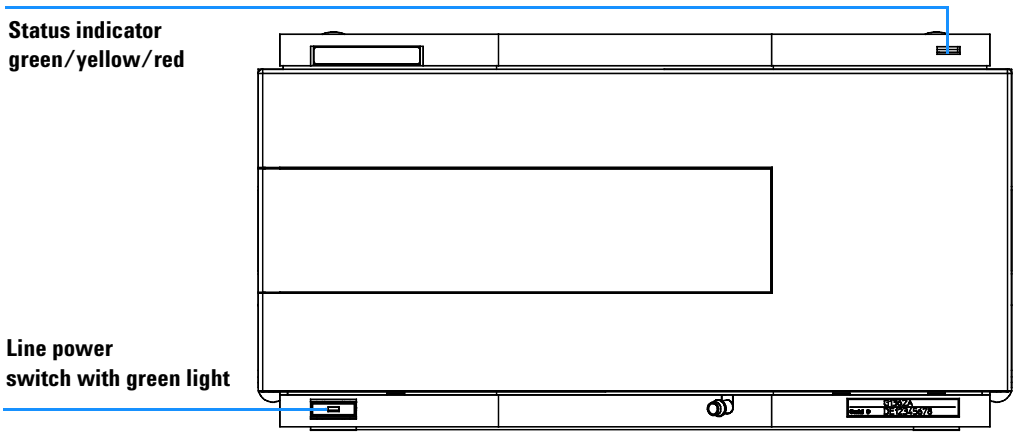


Figure 15 Front View of Detector

4 Connect the power cable to the power connector at the rear of the detector.

5 Connect the CAN cable to other Agilent 1200 Series modules.

6 If a Agilent ChemStation is the controller, connect either

- the GPIB cable to the detector or
- the LAN connection to the LAN interface board in the detector.

7 Connect the analog cable (optional) for a chart recorder, integrator or other data collection device.

8 Connect the APG remote cable (optional) for non-Agilent 1200 Series instruments.

9 Turn ON power by pushing the button at the lower left hand side of the detector. The status LED should be green.

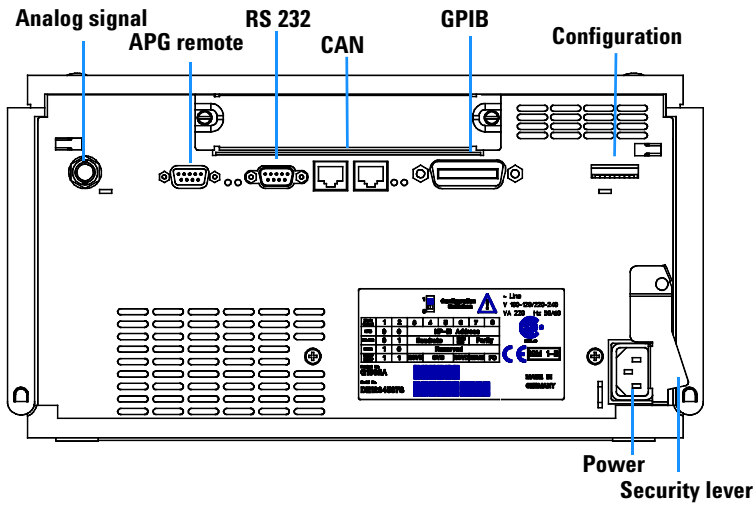


Figure 16 Rear View of Detector

NOTE The detector is turned on when the line power switch is pressed and the green indicator lamp is illuminated. The detector is turned off when the line power switch is protruding and the green light is OFF.

WARNING **To disconnect the detector from line, unplug the power cord. The power supply still uses some power, even if the power switch at the front panel is turned OFF.**

NOTE The detector was shipped with default configuration settings. To change these settings see "Setting the 8-bit Configuration Switch" in the Service Manual.

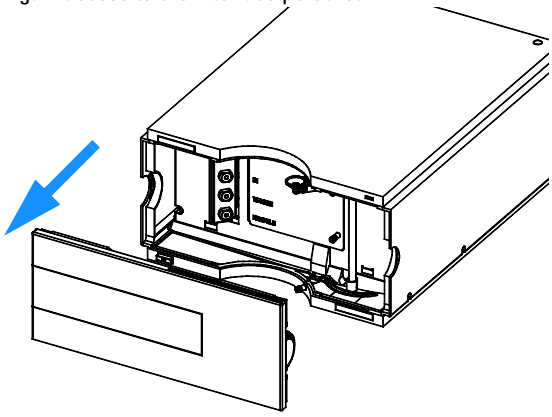
Flow Connections to the Detector

Preparations	Detector is installed in the LC system.
Parts required	Other modules Interface tubing kit G1362-68706 Interfacing capillary G1362-87300 see"Detector Accessory Kit Contents" on page 35 1/4" wrench.

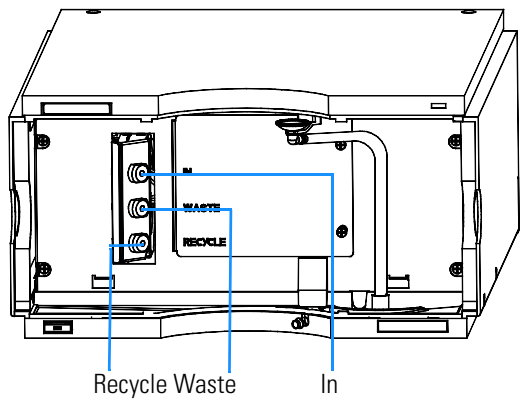
WARNING **When working with solvents please observe appropriate safety procedures (for example, goggles, safety gloves and protective clothing) as described in the material handling and safety data sheet supplied by the solvent vendor, especially when toxic or hazardous solvents are used.**

NOTE The flow cell is shipped with a filling of isopropanol (also recommended when the instrument and/or flow cell is shipped to another location). This is to avoid breakage due to subambient conditions.

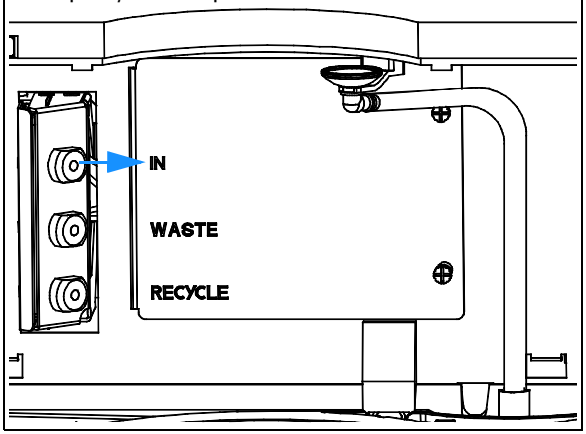
1 Press the release buttons and remove the front cover to gain access to the interface port area.



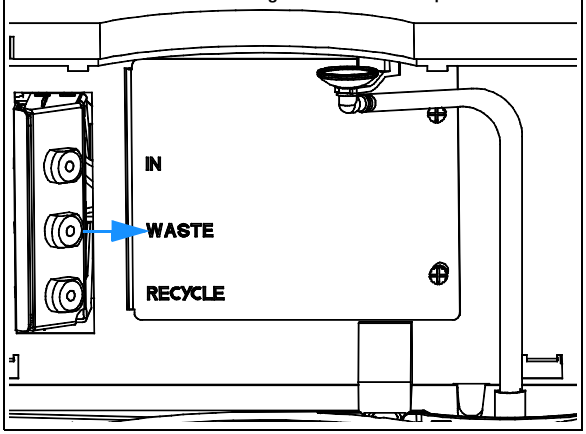
2 Locate the in, waste and recycle ports.



3 Remove the blanking plug and connect the interfacing capillary to the in port.



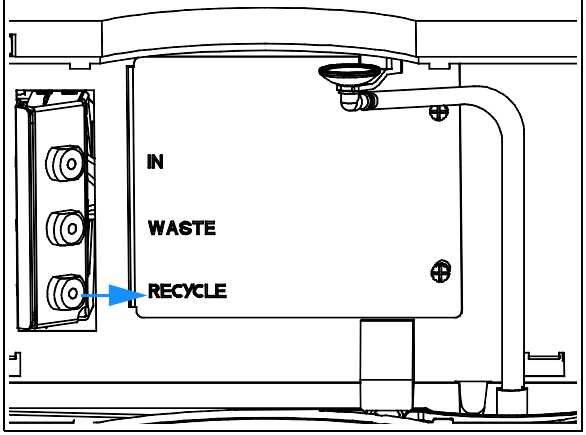
4 Remove the blanking plug and connect one of the tubes from the interface tubing kit to the waste port.



NOTE

The back pressure rating of the refractive index flow cell is 5 bar. Therefore the RI detector must be the last module in the flow path. If an additional detector is to be installed it must be connected upstream of the refractive index detector in order to avoid damage to the RID flow cell due to overpressure.

5 Remove the blanking plug and connect the other tube from the interface tubing kit to the recycle port.



NOTE

Remove all blanking plugs from all outlet ports (waste & recycle) of the detector to avoid potential damage to the flow cell, if the recycle valve is accidentally switched to one of these ports, while flow is applied to the detector.

6 Direct the waste tube into a suitable waste container. Make sure that there is no restriction of this tube.

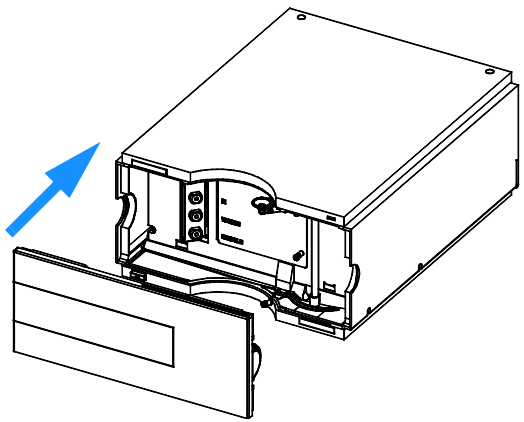
7 If solvent recycling is to be used direct the recycle tube into the solvent bottle. Make sure that there is no restriction of this tube.

NOTE

To optimize detector performance the waste container and solvent bottle should be positioned above the level of the refractive index detector and solvent pump (e.g. in the solvent compartment). This will maintain a *slight* pressure in the sample cell. Route the tubing behind the front covers of the Agilent 1200 Series modules in the stack.

8 Establish flow and observe if leaks occur.

9 Replace the front cover.



The installation of the detector is now complete.

4
Using the Refractive Index Detector

Before Using the System 46
Refractive Index Detector Control 47
Refractive Index Detector Settings 49
Refractive Index Detector More Settings 51
Running a Checkout Sample 54
Checking Baseline Noise and Drift 59

This chapter provides information on how to set up the detector for an analysis and explains the basic settings.

Operation of the Refractive Index Detector

This chapter can be used for

- preparing the system,
- to learn the set up of an HPLC analysis and
- to use it as an instrument check to demonstrate that all modules of the system are correctly installed and connected. It is not a test of the instrument performance.
- Learn about special settings

Before Using the System

Solvent Information

Observe recommendations on the use of solvents in chapter “Solvents” in the pump’s reference manual.

Priming and Purging the System

When the solvents have been exchanged or the pumping system has been turned off for a certain time (for example, overnight) oxygen will re-diffuse into the solvent channel between the solvent reservoir, vacuum degasser (when available in the system) and the pump. Solvents containing volatile ingredients will slightly lose these. Therefore priming of the pumping system is required before starting an application.

Table 5 Choice of Priming Solvents for Different Purposes

Activity	Solvent	Comments
After an installation	Isopropanol	Best solvent to flush air out of the system
When switching between reverse phase and normal phase (both times)	Isopropanol	Best solvent to flush air out of the system
After an installation	Ethanol or Methanol	Alternative to Isopropanol (second choice) if no Isopropanol is available
To clean the system when using buffers	Bidistilled water	Best solvent to re-dissolve buffer crystals
After a solvent change	Bidistilled water	Best solvent to re-dissolve buffer crystals
After the installation of normal phase seals (P/N 0905-1420)	Hexane + 5% Isopropanol	Good wetting properties

1 Open the purge valve of your pump (by turning it counterclockwise) and set flow rate to 3-5 ml/min.

2 Flush all tubes with at least 30 ml of solvent.

3 Set flow to required value of your application and close the purge valve.

Pump for approximately 30 minutes before starting your application (for some solvents an even longer flush and equilibration time might be needed).

Refractive Index Detector Control

The following operating instructions were generated using the Agilent B.01.03 ChemStation as operating software.

How To Get There:

The Agilent 1200 RID Control dialog box is displayed when you select **More RID...** from the Instrument menu (**More RID...** is available in **Full Menus** only) and select **Control**... from the **More RID...** submenu.

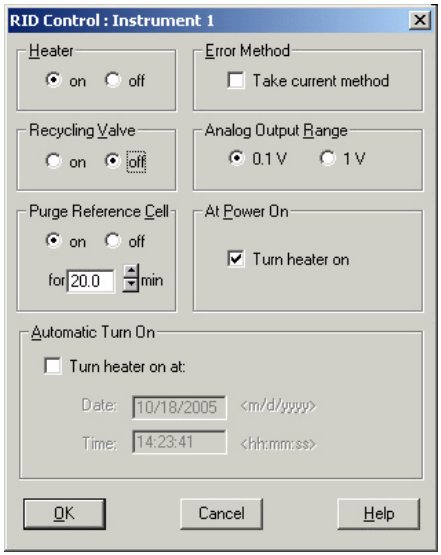


Figure 17 Refractive Index Detector Control

- **Heater:** Select the On option to switch the RID heater on. This parameter requires to set the Optical Unit Temperature.
  Select the Off option to switch the optical unit heater off.
- **Error Method:** The Error Method group enables you to select the method that is run when an error occurs. It ensures that the instrument shuts down in a controlled manner if the ChemStation control is discontinued for any reason. When Take Current Method is checked the current method is copied to the module and stored; if an error occurs, the module will run the stored method.
- **Recycling Valve:** Select the On option to switch the recycling of the eluent on. The Off option diverts the flow of the Agilent 1200 RID to the waste bottle.
- **Analog Output Range:** The Analog Output Range group allows you to select the voltage ranges of the analog output of the refractive index detector.
  Select 0.1 V to set the full-scale output to 0.1 volts.
  Select 1 V to set the full-scale output to 1 volt.

- **Purge Reference Cell:** This parameter is used to exchange the content of the reference cell in the case of solvent change or reference cell contamination. Enter a time interval (minutes) to purge the reference cell of the Agilent 1200 RID. This will be started immediately if you click OK on this window. Allow additional time for baseline stabilization after purging.
- **At Power On:** The At Power On group allows you to select to turn on the Agilent 1200 RID temperature controller when the Agilent 1200 RID is switched on.
- **Automatic Turn On:** You can set a date and time at which the temperature controller is switched on automatically.
  Select Turn Agilent 1200 RID on at: to activate the date and time fields, and enter the date and time in the appropriate fields in the specified format.

Refractive Index Detector Settings

The following operating instructions were generated using the Agilent B.01.03 ChemStation as operating software.

How To Get There:

The Agilent 1200 RID Signal dialog box is displayed when you select **Setup RID Signal** from the **Instrument menu**.

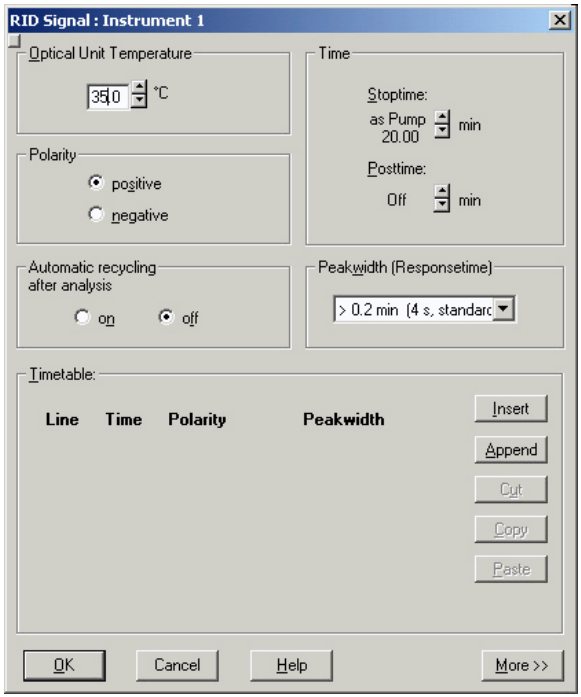


Figure 18 Refractive Index Detector Settings

- **Optical Unit Temperature:** This item sets the temperature of the optical unit. The optical unit of the Agilent 1200 RID can be operated between 5°C above ambient and 55°C. The recommended setting is 5°C above ambient. This will improve baseline stability.
- **Polarity:** This item sets the polarity of the RID signal. Because of the nature of analytes and eluents refractive index detectors can show negative and positive peaks, even within a run. Select the Signal Polarity you expect from your data from Negative or Positive.
- **Automatic Recycling:** This parameter can be used to select between automatic recycling of the eluent (on) or directing the eluent to the waste outlet of the RID (off) after the run.

- **Time:**
  Stoptime
  Stoptime enables you to set a time at which the RID stops an analysis. If the RID is used with other Agilent 1200 Series modules, the RID stoptime stops the RID only and does not stop any other modules.
  Limits: 0.00 to 99999.00 minutes, asPump (the stoptime of the pump when an Agilent pump is configured), asInj (the stoptime of the injector if an Agilent 1200 injector but no Agilent pump is configured) or noLimit (an infinite run time).
  The stoptime setting depends on the configured pump. If you have an Agilent pump with an Agilent injector, then the pump is the stoptime master (asPump). If you have a non-Agilent pump and an Agilent 1200 injector then the injector is the stoptime master (asInj).

  Posttime
  You can set the Posttime so that your RID remains in the not ready state during the Posttime to delay the start of the next analysis. A Posttime period can be used to allow your column to equilibrate after changes in solvent composition.
  Limits: 0 to 99999.00 minutes or Off. Off sets the posttime to 0.0 min.
- **Peakwidth:** Peakwidth enables you to select the peak width (response time) for your analysis. The peak width is defined as the width of a peak, in minutes, at half the peak height. Set the peak width to the narrowest expected peak in your chromatogram. The peak width sets the optimum response time for your RID.
  Limits: When you set the peak width (in minutes), the corresponding response time is set automatically and the appropriate data rate for signal acquisition is selected (please refer to the ChemStation's Online help for more details).

Refractive Index Detector More Settings

The following operating instructions were generated using the Agilent B.01.03 ChemStation as operating software.

How To Get There:

The Agilent 1200 RID Signal dialog box is displayed when you select **Setup RID signal** from the **Instrument menu**. The **More Button** displays additional Menus.

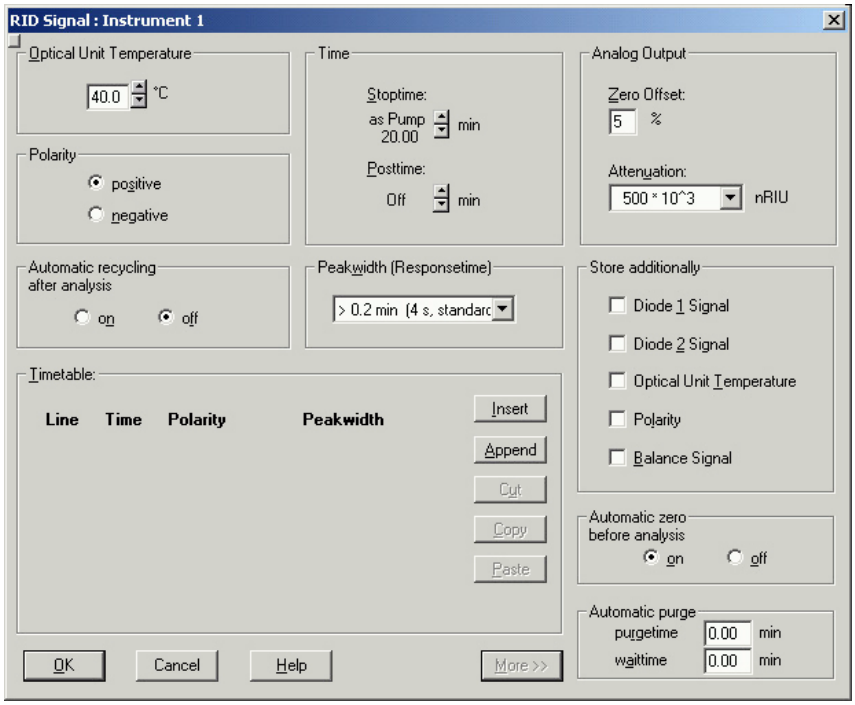


Figure 19 More RID Settings

- **Analog Output:** If the Analog Output is used a zero offset (limits between 1 and 99%) can be selected to enable the display of negative peaks. The attenuation settings helps to keep all peaks on scale. Choose the appropriate setting from the list.

- **Store Additionally:** Here you can choose to store additional signal that may help during method development and diagnosis with the Agilent 1200 RID. The following parameters can be selected:

  Diode 1 signal
  The RID signal is based on the ratio of the light level that is measured by two photodiodes. The RID signal is zero if the two diodes show the same light level. This parameter allows you to store individually the signal measured by the diode 1.

  Diode 2 signal
  The RID signal is based on the ratio of the light level that is measured by two photodiodes. The RID signal is zero if the two diodes show the same light level. This parameter allows you to store individually the signal measured by the diode 2.

  Optical unit temperature
  This parameter activates the storage of the optical unit temperature signal.

  Polarity
  This parameter activates the storage of polarity switching during the run.

  Balance signal
  This parameter activates storage of the diode balance signal during a run. This helps to diagnose peaks that exceed the dynamic range of the RID, for example in the case of extremely high concentrations/signals.

- **Automatic Zero:** This setting allows you to activate an automatic zeroing of the signal before the run is started. If automatic purge is selected, the purge will be performed before the automatic zero.
- **Automatic Purge:** This parameter can be used to do a purge of the reference cell and wait additional time for baseline stabilization. It will be initiated each time when the run is started. This should only be used if the content of the reference cell is expected to degrade during a run. The automatic purge will be finished before the autozero is performed and before the injection is done.

Running a Checkout Sample

This chapter describes the check out of the Agilent 1200 Series refractive index detector using the Agilent isocratic checkout sample.

When required	If you want to checkout the detector
Hardware required	LC system with G1362A RID
Parts required	Zorbax Eclipse XDB C18, 150mm x 4.6mm i.d. (Part number 993967-902) Agilent isocratic checkout sample (Part number 01080-68704)

Starting Your Detector

1 Turn **ON** the detector.

2 You are now ready to change the settings of your detector.

Setting the Chromatographic Conditions

1 Set up the instrument with the following chromatographic conditions.

Table 6 Chromatographic Conditions

Mobile phases	30% Water, 70% Acetonitrile
Column	Zorbax Eclipse XDB C18, 150mm x 4.6mm i.d.
Sample	Isocratic standard sample
Flow rate	1.5 ml/min
Stroke A	20 µl
Stop time	10 minutes
Injection volume	20 µl
Column compartment temperature	25 ° C
Optical unit Temperature	35 ° C
Polarity	Positive
Peak Width (Response time)	0.2 minutes (4 seconds, standard)

2 Set the RID setpoints according to Figure 20.

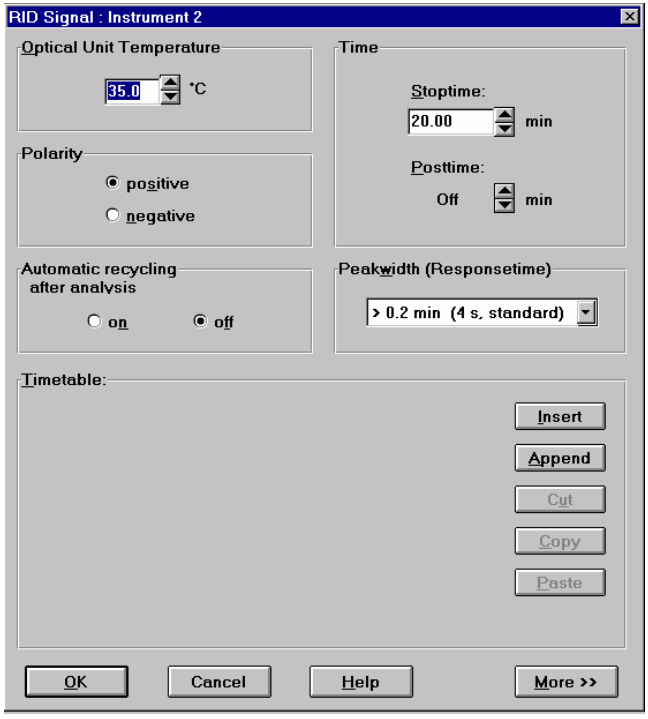


Figure 20 RID Check Out Sample Parameters

3 Turn the heater **ON** and purge the detector reference cell for 20 minutes as shown in Figure 21:

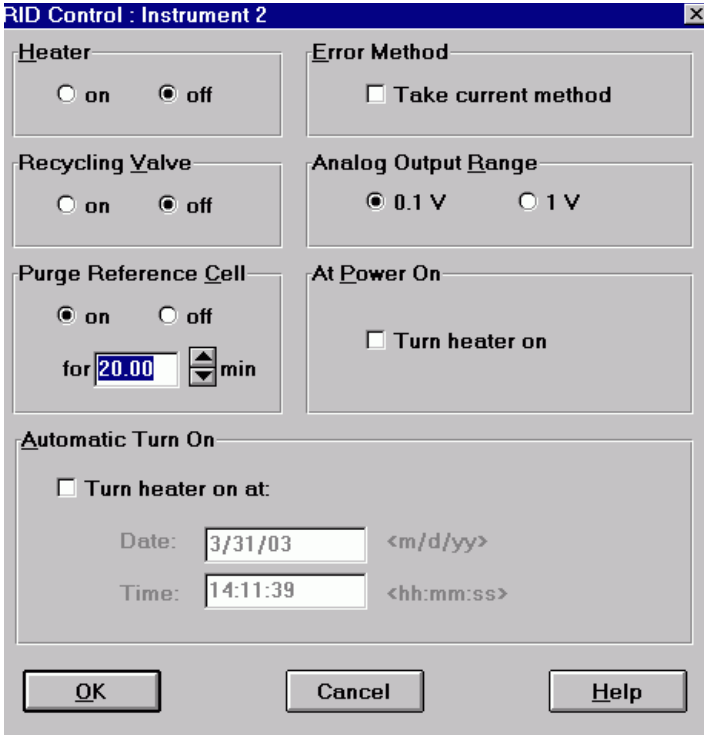


Figure 21 RID Check Out Sample Control

4 When purging has finished allow the baseline to stabilize and start the analysis.

5 If you are using the Control Module in place of the Agilent ChemStation enter the **Plot** function and select **Ref.Index**, set a **time range** of 10 minutes and the **RID signal** from -4000 to 40000 nRIU.

NOTE

The resulting chromatogram should only be seen as a qualitative example, the checkout procedure is not meant as a quantitative procedure. Its intent is only to verify the presence of the for peaks from the checkout sample - nothing more.

Please be aware of the large negative air / solvent peak from the injection (cut out from the bottom of the following figure) prior to the first peak of interest. This is to be expected in a regular chromatogram, especially if a non-degassed sample is injected into degassed solvent and the sample solvent properties don't perfectly match with the mobile phase properties. Only similar zooming factors in the display of a chromatogram will lead to similar looking results.

The resulting chromatogram is shown in Figure 22:

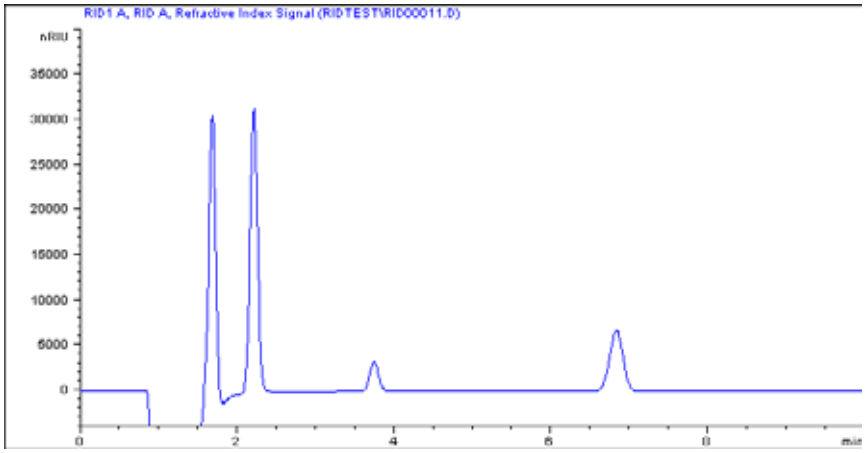


Figure 22 Isocratic Standard Sample Chromatogram

Checking Baseline Noise and Drift

This chapter describes checking the baseline noise and drift for the Agilent 1200 Series refractive index detector.

When required	If you want to checkout the detector
Hardware required	LC system with G1362A RID
Parts required	Restriction capillary (Part number G1362-87301)

Starting Your Detector

1 Turn **ON** the detector.

2 You are now ready to change the settings of your detector.

Setting the Test Conditions

1 Connect the restriction capillary directly between the column compartment heat exchanger outlet and the in port of the detector.

2 Set up the instrument with the following test conditions.

Table 7 Chromatographic Conditions

Mobile phases	LC grade water
Column	Restriction capillary 2.7m x 0.17mm i.d.
Flow rate	1.0 ml/min
Compressibility	46
Stroke	20 µl
Stop time	20 minutes
Column compartment temperature	40 ° C
Optical unit Temperature	40 ° C
Polarity	Positive
Peak Width (Response time)	0.2 minutes (4 seconds, standard)

3 Set the RID setpoints according to Figure 23.

NOTE

The optical unit temperature must be set at least 5 ° C above ambient conditions. Therefore if ambient temperature is above 30 ° C higher values for Optical unit Temperature and Column compartment temperature must be set.

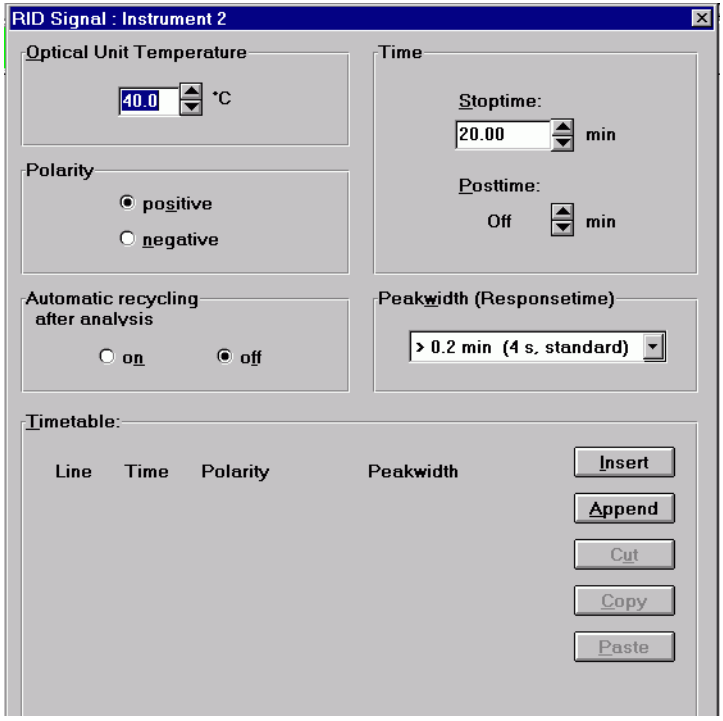


Figure 23 RID Baseline Check Parameters

NOTE The Agilent ChemStation can automatically calculate the baseline short term noise, long term noise (wander) and drift. Follow steps 4 to 9.

NOTE If you are not using the Agilent ChemStation go to step 10.

4 Edit the Agilent ChemStation method (**Method and Run Control- Method Edit Entire Method**).

5 Specify the report style Performance + Noise as shown in Figure 24

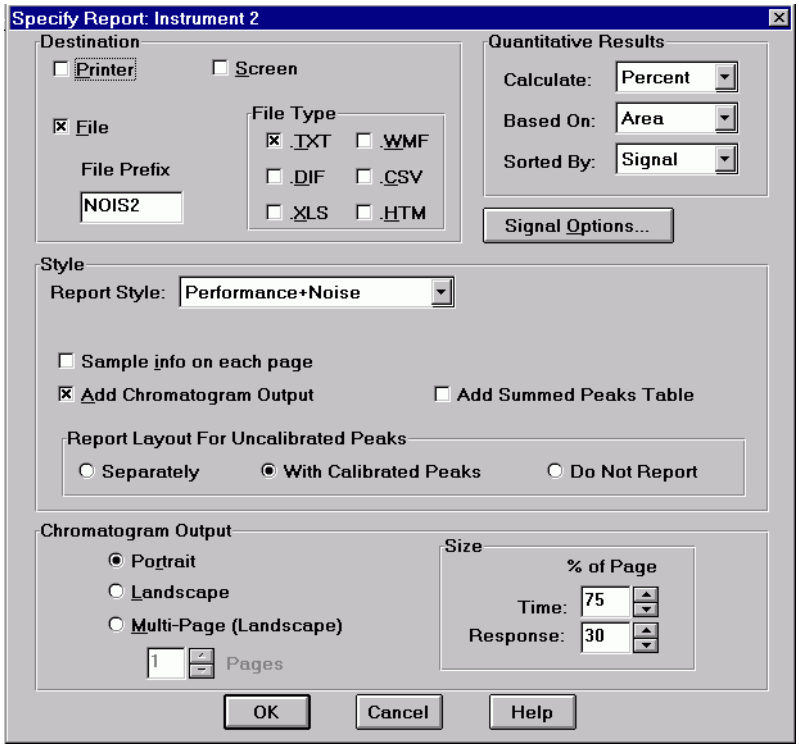


Figure 24 RID Baseline Check Out Report

6 Set the noise determination time range to 0 - 20 minutes as shown in Figure 25:

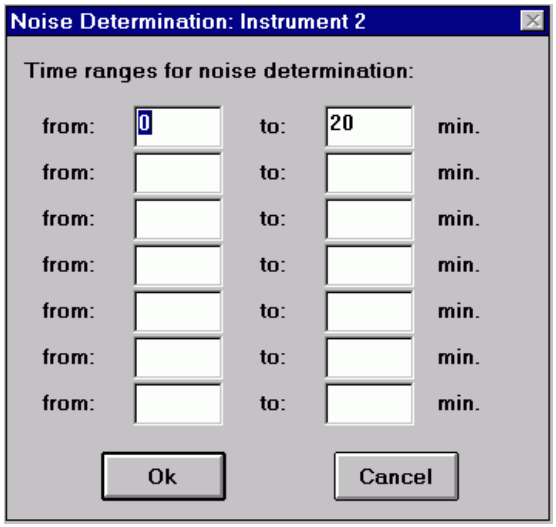


Figure 25 RID Baseline Check Out Noise Ranges

7 **Save** the Agilent ChemStation method.

8 Turn the heater **ON** and purge the detector reference cell for 20 minutes as shown in Figure 26:

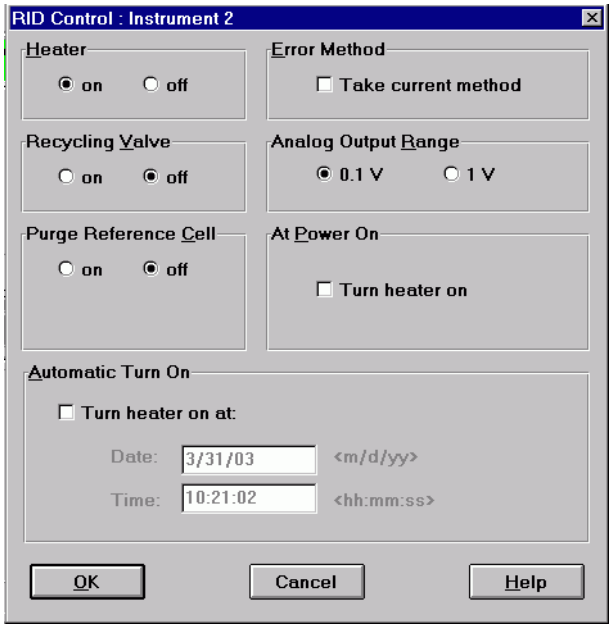
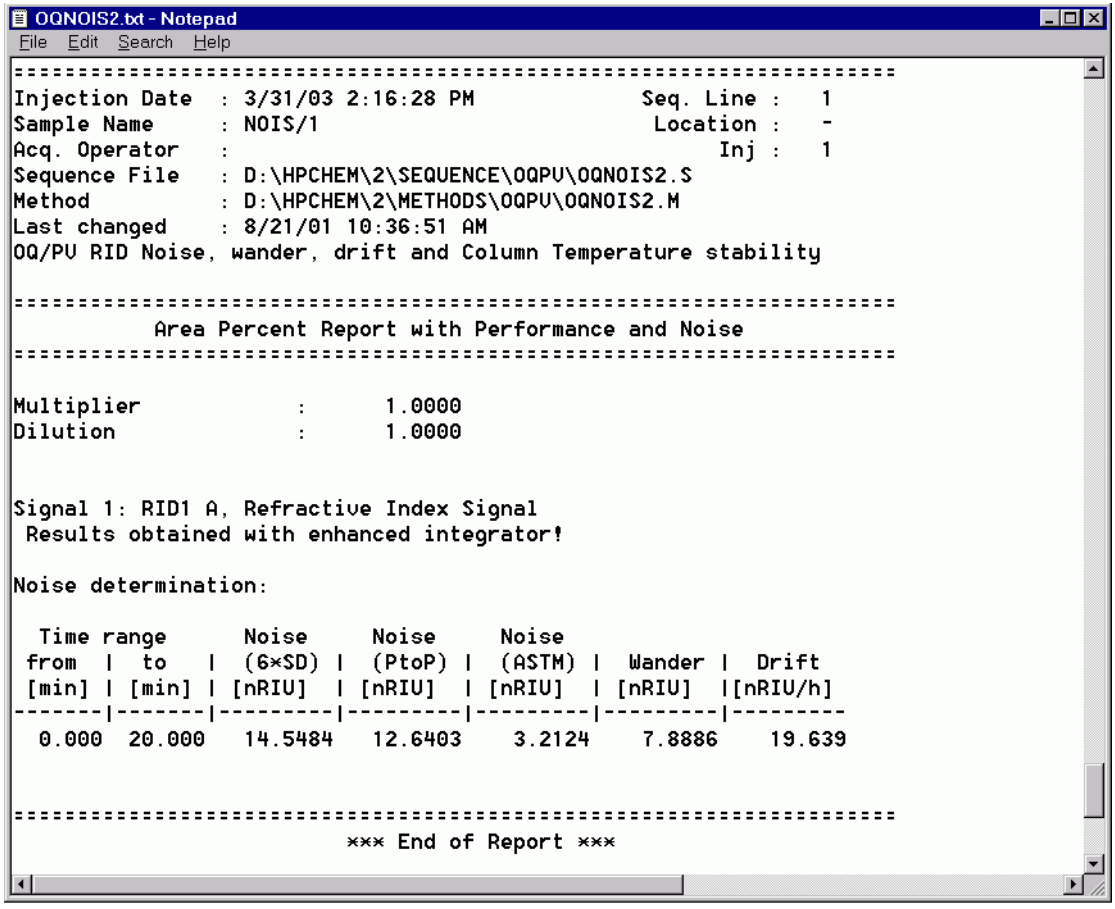


Figure 26 RID Baseline Check Control

9 When purging has finished allow the baseline to stabilize and start the sequence (blank run - no injection).

10 If you are using the Control Module in place of the Agilent ChemStation enter the **Plot** function and select **Ref.Index**, set a **time range** of 20 minutes and the **RID signal** from -200 to 200 nRIU.

11 The Agilent ChemStation report in shown in Figure 27:



OQNOIS2.txt - Notepad

File Edit Search Help

```
================================================================================
Injection Date  : 3/31/03 2:16:28 PM             Seq. Line :   1
Sample Name     : NOIS/1                          Location :   -
Acq. Operator   :                                     Inj :   1
Sequence File   : D:\HPCHEM\2\SEQUENCE\OQPU\OQNOIS2.S
Method          : D:\HPCHEM\2\METHODS\OQPU\OQNOIS2.M
Last changed    : 8/21/01 10:36:51 AM
OQ/PU RID Noise, wander, drift and Column Temperature stability

================================================================================
           Area Percent Report with Performance and Noise
================================================================================

Multiplier             :      1.0000
Dilution               :      1.0000


Signal 1: RID1 A, Refractive Index Signal
 Results obtained with enhanced integrator!

Noise determination:

  Time range      Noise     Noise     Noise
 from  |  to   |  (6*SD) |  (PtoP) |  (ASTM) |  Wander |  Drift
 [min] | [min] | [nRIU]  | [nRIU]  | [nRIU]  | [nRIU]  |[nRIU/h]
-------|-------|---------|---------|---------|---------|---------
  0.000  20.000   14.5484   12.6403    3.2124    7.8886    19.639


================================================================================
                          *** End of Report ***
```

Figure 27 Baseline Check Out Results

Evaluation

For the Control Module *Rescale* the plot and measure the baseline noise and drift on the screen. If a printer is configured for the Agilent 1200 Series instrument the plot can be printed by pressing the **m** key and selecting **Print Plot**.

The following values are calculated automatically by the Agilent ChemStation.

- *Noise (ASTM):* The short term noise in nRIU based on ASTM method E-1303-95 “Practice for Refractive Index Detectors used in Liquid Chromatography” using 0.5 minute segments.
- *Wander:* The long term noise in nRIU based on ASTM method E-1303-95 “Practice for Refractive Index Detectors used in Liquid Chromatography” using 0.5 minute segments.
- *Drift:* The drift in nRIU/hour based on ASTM method E-1303-95 “Practice for Refractive Index Detectors used in Liquid Chromatography” measured over 20 minutes.

NOTE Factors that will affect the baseline stability include;

- Variations in the optics or eluent temperature
- Pressure fluctuations in the sample cell
- The quality of the water used
- Air bubbles in the flow cell

See “Refractive Index Detector Control” on page 47.

5 Optimizing the Refractive Index Detector

Refractive Index Detector Optimization 68
Potential Causes for Baseline Problems 70
Detector Equilibration 71

This chapter provides information on how to optimize the detector.

Refractive Index Detector Optimization

Follow these thirteen points to optimize the performance of your refractive index detector.

1 Position the solvent and waste reservoirs correctly

Position the solvent and waste reservoirs above the level of the refractive index detector and solvent pump. This maintains a slight pressure in the sample cell and will improve the performance of the detector.

2 Do not overpressurize the flow cell

Be aware to not exceed a 5 bar pressure drop after the flow cell when hooking up additional devices like other detectors or a fraction collector. If an additional detector is installed place upstream in the flow path it before the G1362A refractive index detector.

3 Use the correct solvents

To minimize baseline noise and drift solvents must be LC grade and filtered prior to use.

4 Check for leaks

Leaks within the LC instrument that the refractive index detector is connected to will cause problems with baseline long term noise or drift. Confirm that the instrument is free from leaks by performing the Agilent 1200 Series diagnostic pressure test (for the high pressure parts of the system between pump and column). Ensure that the connections from the on-line vacuum degasser to the pump and the detector inlet, waste and recycle connections are air tight.

5 Verify frit, filter and fitting quality

Partially blocked frits, filters and fittings can cause baseline long term noise. Verify that the pressure drop across all such parts is within expected limits.

6 Control the optical unit temperature

Always control the optical unit temperature (heater = ON) for maximum detector sensitivity or with samples that could precipitate in the sample cell at room temperature and set an elevated optical unit temperature at least 5 °C above ambient conditions.

7 Use an appropriate response time

For most applications a setting of 4 seconds is adequate. Only for high speed analyses (short columns at high flow rates) a lower setting is recommended. Bear in mind that even if the response time setting is too high fast peaks will appear a little smaller and broader but retention time and peak areas are still correct and reproducible.

8 Recycle mobile phase

Use the recycle valve to allow automatic recycling of mobile phase delivered when no analysis is running. The pump flow can therefore continue uninterrupted until the next analysis without wasting mobile phase solvents. In addition the refractive index detector is always stabilized and ready for immediate use.

9 Consider using a degasser
For many solvents you can achieve better baseline stability, when using a degasser. For some solvents a degasser might not lead to a better baseline quality.

10 Flush the degasser

If flow is stopped and mobile phase remains inside the on-line vacuum degasser the solvent composition will change. When re-starting the flow or when using new mobile phase flush each degasser channel used for 10 minutes at the maximum flow rate of the pump (with the purge valve of the pump open to avoid a potential over-pressure in the RI detector's flow cell).

11 Use pre-mixed solvents, only
Don't use a pump for mixing solvents. When operating the RI detector together with a quaternary pump, bypass the MCGV in the quaternary pump. You have to virtually convert the quaternary pump into an isocratic one, by directly connecting the solvent inlet tubing from degasser or solvent bottle to the active inlet valve of the pump (use adapter 0100-1847, which is delivered with the accessory kit of the detector).

12 Consider solvent changes with time

Baseline drift can be caused by the tendency of certain solvents to change over time. For example the acetonitrile content of acetonitrile/water mixtures will decrease, tetrahydrofuran will form peroxides, the amount of water in hygroscopic organic solvents will increase and solvents such as tetrahydrofuran held in the reference cell may begin to regas.

13 Eliminate mobile phase/column combination problems

Certain mobile phases in combination with specific columns can generate long term baseline noise. For example acetonitrile/water mobile phases with certain aminopropyl bonded phase columns. To eliminate the combination of mobile phase and column as a cause of long term noise replace the column with the restriction capillary (G1362-87301) and re-evaluate the detector performance.

Potential Causes for Baseline Problems

Noise (short term)

Typically the sources for short term noise are either electronic (check the settings for the peak widths, check for ambient sources of electronic noise) or they are related to the solvents, their composition and flow (in order to verify this, turn off the pump, consider degassing your solvents, use only premixed solvents).

Wander (long term noise)

Excessive wander is an indication for a general system or environmental instability (system or laboratory might not be thermally stable, control instrument and laboratory temperature). Verify that the solvent properties are constant over time (flush out contamination, use only stabilized and premixed solvents). Clean the parts in the flow path and allow the system to be flushed out and equilibrated.

Drift

Excessive drift is an indication for a general system or environmental instability (system or laboratory might not be thermally stable, control instrument and laboratory temperature). Verify that the solvent properties are

constant over time (flush out of contamination, use only stabilized solvents). Clean parts in the flow path and allow the system to be flushed out and equilibrated.

Detector Equilibration

The Refractive Index (RI) is a function of temperature, pressure and a property of the used solvent (it changes with solvent composition, degassing level and due to any trace of contamination). Therefore the Refractive Index Detector will detect any change in any of these parameters as a change in its signal and a variation of its baseline. Therefore the detector will trace down any instabilities in the system and the environment as well. It may sometimes appear, as if the detector itself was unstable or generating an unstable baseline, where in fact, the detector is simply displaying the instabilities of the environment and the rest of the system. By this the detector is often - without justification - blamed for instabilities, which it does not generate itself, but only detect. The fact that this detector is a universal detector makes it also sensitive to instabilities introduced to it from outside the detector.

This makes it very important to have a very stable environment and system for achieving best possible baseline stability. The baseline will get the better, the longer the system is used under identical and stable conditions. Keep the temperature in your laboratory and system constant and controlled. Ideally a system with an RID should be used always with the same type of analysis (stable solvent composition, temperature, flow rates, do not switch the pump off after analysis, instead just recycle solvents or at least reduce only the flow. Switch valves and settings only when needed. Do not expose the detector to draft of air or to vibrations). A change of any of these parameters may require a considerable amount of time for re-equilibration.

6
Troubleshooting Overview

Overview of the Detector's Indicators and Test Functions 74
Status Indicators 75

This chapter gives an overview about the troubleshooting and diagnostic features.

Overview of the Detector's Indicators and Test Functions

Status Indicators

The detector is provided with two status indicators which indicate the operational state (prerun ready, not-ready, run, and error states) of the detector. The status indicators provide a quick visual check of the operation of the detector (see "Status Indicators" on page 75).

Error Messages

In the event of an electronic, mechanical or hydraulic failure, the detector generates an error message in the user interface. For each message, a short description of the failure, a list of probable causes of the problem, and a list of suggested actions to fix the problem are provided (see "Error Messages" on page 80).

Not-Ready Messages

During the wait for a specific condition to be reached or completed the detector will generate a not-ready message. For each message a short description is provided (see "Not-Ready Messages" in the Service Manual).

Refractive Index Calibration

Refractive index calibration is recommended after exchange of the optical unit to ensure correct operation of the detector. The procedure uses a solution of known refractive index compared to LC grade water (see "Refractive Index Calibration" in the Service Manual).

Optical Balance

Optical balance allows the balance of light falling on the light receiving diodes to be restored. The sample and reference cells must both be fully purged before the procedure is started, see "Optical Balance" in the Service Manual).

Status Indicators

Two status indicators are located on the front of the detector. The lower left indicates the power supply status, the upper right indicates the detector status.

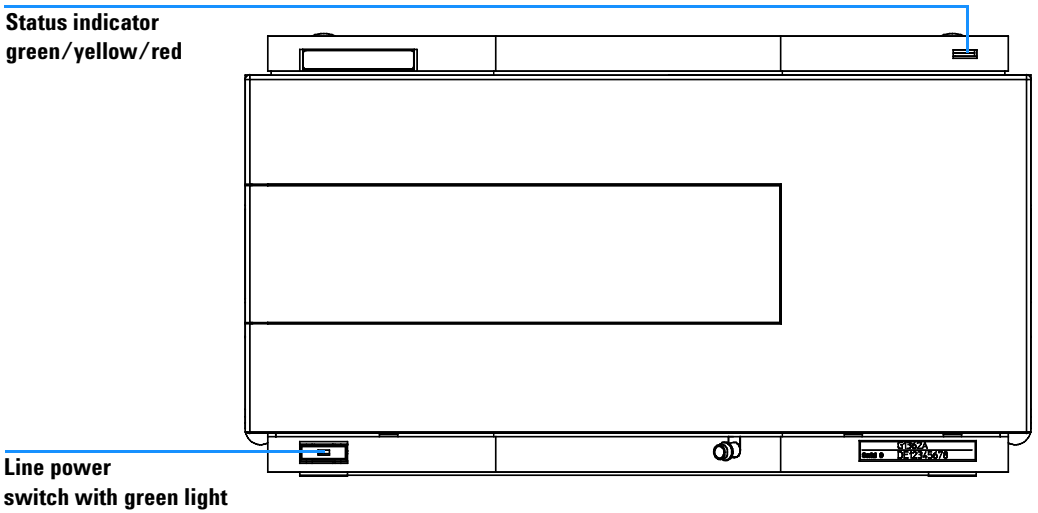


Figure 28 Location of Status Indicators

Power Supply Indicator

The power supply indicator is integrated into the main power switch. When the indicator is illuminated (*green*) the power is ON.

Detector Status Indicator

The detector status indicator indicates one of four possible detector conditions:

- When the status indicator is OFF (and power switch light is on), the detector is in a *prerun* condition, and is ready to begin an analysis.
- A *green* status indicator, indicates the detector operating in a running analysis.
- A *yellow* indicator indicates a *not-ready* condition. The detector is in a not-ready state when it is waiting for a specific condition to be reached or completed, for example, immediately after changing a set point.
- An *error* condition is indicated when the status indicator is *red*. An error condition indicates the detector has detected an internal problem which affects correct operation of the detector. Usually, an error condition requires attention (e.g. leak, defective internal components). An error condition always interrupts the analysis.

7
Repairing the Refractive Index Detector

Introduction to the Repairing the Refractive Index Detector 78

This chapter provides general information on repairing the detector.

Introduction to the Repairing the Refractive Index Detector

Maintenance

The detector is designed for easy repair. The most frequent repairs such as flow cell flushing can be done from the front of the detector with the detector in place in the system stack. These repairs are described in "Detector Maintenance Procedures" on page 82.

Exchanging Internal Parts

Some repairs may require exchange of defective internal parts. Exchange of these parts requires the removal of the detector from the stack, removing the covers, and disassembling the detector. The security lever at the power input socket prevents the detector cover from being removed when line power is still connected. These repairs are described in "Exchanging Internal Parts" in the Service Manual.

WARNING **To prevent personal injury, the power cable must be removed from the instrument before opening the detector cover. Do not connect the power cable to the detector while the covers are removed.**

WARNING **To prevent personal injury, be careful, when getting in contact with sharp metal edges.**

WARNING **When working with solvents please observe appropriate safety procedures (for example, goggles, safety gloves and protective clothing) as described in the material handling and safety data sheet supplied by the solvent vendor, especially when toxic or hazardous solvents are used.**

CAUTION Electronic boards and components are sensitive to electronic discharge (ESD). In order to prevent damage always use an ESD protection (for example, the ESD wrist strap from the accessory kit of another Agilent 1200 Series module) when handling electronic boards and components (see "Using the ESD Strap" on page 80).

CAUTION There is a risk of damaging hardware due to overheating when operating the instrument without covers.

Cleaning the Detector

The detector case should be kept clean. Cleaning should be done with a soft cloth slightly dampened with water or a solution of water and mild detergent. Do not use an excessively damp cloth allowing liquid to drip into the detector.

WARNING **Do not let liquid drip into the detector. It could cause shock hazard and it could damage the detector.**

Using the ESD Strap

Electronic boards are sensitive to electronic discharge (ESD). In order to prevent damage, always use an ESD strap supplied in the standard accessory kit of other Agilent 1200 Series modules when handling electronic boards and components.

1. Unwrap the first two folds of the band and wrap the exposed adhesive side firmly around your wrist.
2. Unroll the rest of the band and peel the liner from the copper foil at the opposite end.
3. Attach the copper foil to a convenient and exposed electrical ground.

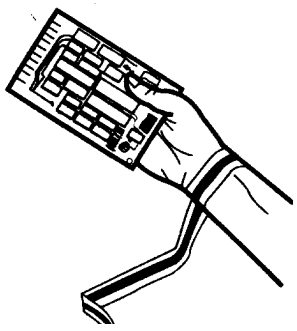


Figure 29 Using the ESD Strap

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Maintaining the Refractive Index Detector

Detector Maintenance Procedures 82
Flow Cell Flushing 83
Correcting Leaks 84
Replacing Leak Handling System Parts 85
Replacing the Detector's Firmware 86

This chapter describes the maintenance of the detector.

Detector Maintenance Procedures

On the following pages maintenenance procedures are described that can be carried out without opening the main cover.

Table 8 Maintenance Procedures

Procedure	Typical Frequency	Notes
Flow cell flushing	If flow cell is contaminated.	
Leak sensor drying	If leak has occurred.	Check for leaks.
Leak handling System replacement	f broken or corroded.	Check for leaks.
Replacing the detector's Firmware	If not up to date or corrupted.	

Flow Cell Flushing

When required	If flow cell is contaminated
Tools required	Glass syringe, adapter
Parts required	Strong solvent, tubings to waste

NOTE Aqueous solvents in the flow cell can build up algae. Therefore do not leave aqueous solvents in the flow cell for long periods. Add a small percentage of organic solvents (e.g. Acetonitrile or Methanol ~5%).

NOTE The strong solvent should dissolve any potential contaminants in the flow cell. For example water for aqueous mobile phase buffers, chloroform or tetrahydrofuran for not water soluble contaminants.

In case the cell is contaminated, follow the procedure below.

Flushing Procedure

1 Flush with the strong solvent.

2 Leave this solution in the cell for about one hour.

3 Flush with mobile phase.

CAUTION The strong solvent used may be dangerous and proper attention to safety should be given.

NOTE Do not exceed the flow cell pressure limit of 5 bar (0.5 MPa).

Correcting Leaks

When required	If a leakage has occurred in the valve area or at the capillary connections
Tools required	Tissue Two 1/4 inch wrenches for capillary connections
Parts required	None

1 Remove the front cover.

2 Open the service door

3 Use tissue to dry the leak sensor area and the leak pan.

4 Observe the interface ports and the valve area for leaks and correct, if required.

5 Close the service door.

6 Replace the front cover.

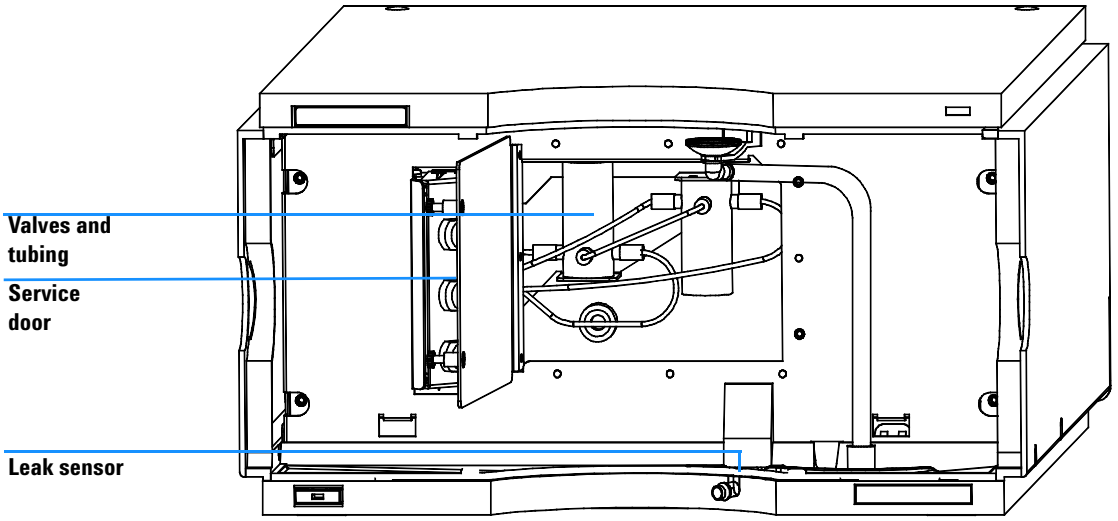

Figure 30 Observing for Leaks

Replacing Leak Handling System Parts

When required	If the parts are corroded or broken
Tools required	None
Parts required	Leak funnel 5061-8388 Leak funnel holder 5041-8389 Leak tubing (120mm) 0890-1711

1 Remove the front cover.
2 Pull the leak funnel out of the leak funnel holder.
3 Pull out the leak funnel with the tubing.
4 Insert the leak funnel with the tubing in its position.
5 Insert the leak funnel into the leak funnel holder.
6 Replace the front cover.

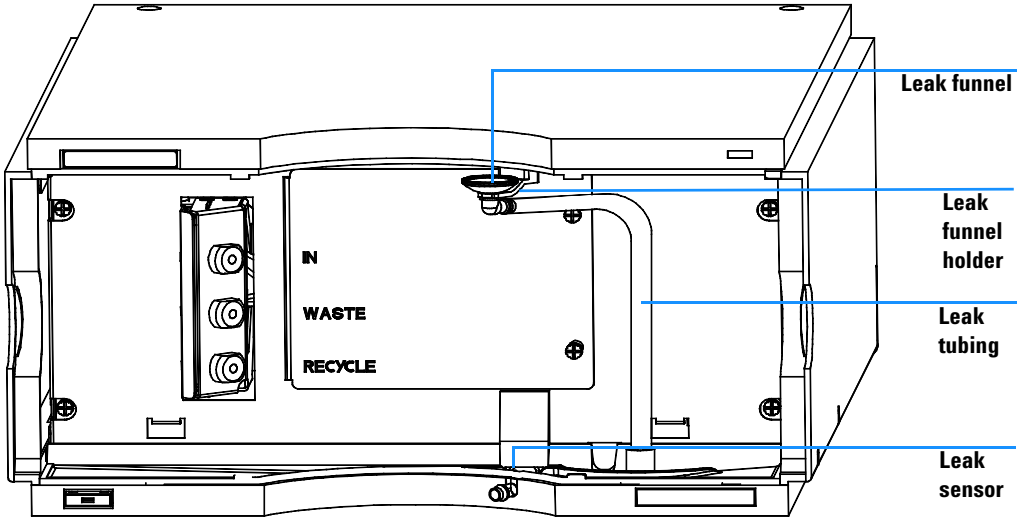

Figure 31 Replacing Leak Handling System Parts

Replacing the Detector's Firmware

When required	If new version solves problems of currently installed version or after exchange of the detector main board (VWDM) the version on board is older than previous installed one.
Tools required	LAN/RS-232 Firmware Update Tool
Parts required	Firmware, tools and documentation from Agilent web site
Preparations	Read update documentation provided with the Firmware Update Tool.

The installation of *older* firmware might be necessary:

- to keep all systems on the same (validated) revision, or
- if third part control software requires a special version.

To upgrade/downgrade the detector's firmware the following steps have to be performed:

1 Download the module's firmware, the LAN/RS-232 FW Update Tool Version 2.00 or above and the documentation from the Agilent web

http://www.chem.agilent.com/scripts/cag_firmware.asp.

2 Load the firmware into the detector as described in the documentation.

3 If the detector main board was replaced, perform a "Refractive Index Calibration" described in the Service Manual to add the recalibration parameters into the board's memory.

4 If detector main board was replaced, re-enter the serial number information of the module through the user interface as described in the Service Manual.

A
Safety Information

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The Waste Electrical and Electronic Equipment (WEEE) Directive (2002/96/EC) 91
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This chapter provides additional information on safety, legal and web.

General Safety Information

The following general safety precautions must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture, and intended use of the instrument. Aligent Technologies assumes no liability for the customer's failure to comply with these requirements.

General

This is a Safety Class I instrument (provided with terminal for protective earthing) and has been manufactured and tested according to international safety standards.

This instrument is designed and certified as a general purpose laboratory instrument for research and routine application only. It is not certified for in-vitro or medical applications.

Operation

Before applying power, comply with the installation section. Additionally the following must be observed.

Do not remove instrument covers when operating. Before the instrument is switched on, all protective earth terminals, extension cords, auto-transformers, and devices connected to it must be connected to a protective earth via a ground socket. Any interruption of the protective earth grounding will cause a potential shock hazard that could result in serious personal injury. Whenever it is likely that the protection has been impaired, the instrument must be made inoperative and be secured against any intended operation.

Make sure that only fuses with the required rated current and of the specified type (normal blow, time delay, and so on) are used for replacement. The use of repaired fuses and the short-circuiting of fuse holders must be avoided.

CAUTION The operator of this instrument is advised that if the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.

Some adjustments described in the manual, are made with power supplied to the instrument, and protective covers removed. Energy available at many points may, if contacted, result in personal injury.

Any adjustment, maintenance, and repair of the opened instrument under voltage should be avoided as much as possible. When inevitable, this should be carried out by a skilled person who is aware of the hazard involved. Do not attempt internal service or adjustment unless another person, capable of rendering first aid and resuscitation, is present. Do not replace components with power cable connected.

Do not operate the instrument in the presence of flammable gases or fumes. Operation of any electrical instrument in such an environment constitutes a definite safety hazard.

Do not install substitute parts or make any unauthorized modification to the instrument.

Capacitors inside the instrument may still be charged, even though the instrument has been disconnected from its source of supply. Dangerous voltages, capable of causing serious personal injury, are present in this instrument. Use extreme caution when handling, testing and adjusting.

When working with solvents please observe appropriate safety procedures (e.g. goggles, safety gloves and protective clothing) as described in the material handling and safety data sheet by the solvent vendor, especially when toxic or hazardous solvents are used.

Safety Symbols

Table 9 shows safety symbols used on the instrument and in the manuals.

Table 9 Safety Symbols

Symbol	Description
	The apparatus is marked with this symbol when the user should refer to the instruction manual in order to protect risk of harm to the operator and to protect the apparatus against damage.
	Indicates dangerous voltages.
	Indicates a protected ground terminal.
	Indicates eye damage may result from directly viewing the light produced by the deuterium lamp used in this product.

WARNING **A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.**

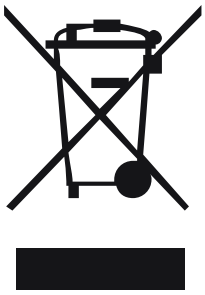
CAUTION A caution alerts you to situations that could cause a possible loss of data. Do not proceed beyond a caution until you have fully understood and met the indicated conditions.

The Waste Electrical and Electronic Equipment (WEEE) Directive (2002/96/EC)

Abstract

The Waste Electrical and Electronic Equipment (WEEE) Directive (2002/96/EC), adopted by EU Commission on 13 February 2003, is introducing producer responsibility on all Electric and Electronic appliances from 13 August 2005.

NOTE



This product complies with the WEEE Directive (2002/96/EC) marking requirements. The affixed label indicates that you must not discard this electrical/electronic product in domestic household waste.

Product Category:
With reference to the equipment types in the WEEE Directive Annex I, this product is classed as a “Monitoring and Control instrumentation” product.

Do not dispose off in domestic household waste

To return unwanted products, contact your local Agilent office, or see www.agilent.com for more information.

Radio Interference

Never use cables other than the ones supplied by Aligent Technologies to ensure proper functionality and compliance with safety or EMC regulations.

Test and Measurement

If test and measurement equipment is operated with equipment unscreened cables and/or used for measurements on open set-ups, the user has to assure that under operating conditions the radio interference limits are still met within the premises.

Sound Emission

Manufacturer's Declaration

This statement is provided to comply with the requirements of the German Sound Emission Directive of 18 January 1991.

This product has a sound pressure emission (at the operator position) < 70 dB.

- Sound Pressure Lp < 70 dB (A)
- At Operator Position
- Normal Operation
- According to ISO 7779:1988/EN 27779/1991 (Type Test)

Solvent Information

Observe the following recommendations on the use of solvents.

Flow Cell

Alkaline solutions (pH > 9.5) dissolve the quartz of a flow cell and should not be left in the system for several days.

Prevent any crystallization of buffer solutions. This will lead into a blockage/damage of the flow cell (maximum allowed pressure on the RID flow-cell is 5.0 bar or 0.5 MPa).

If the flow cell is transported while temperatures are below 5 degree C, it must be assured that the cell is filled with alcohol.

Aqueous solvents in the flow cell can build up algae. Therefore do not leave aqueous solvents sitting in the flow cell. Add small % of organic solvents (e.g. Acetonitrile or Methanol ~5%). Refer to "Flow Cell Flushing" on page 83.

Solvents

Brown glass ware can avoid growth of algae.

Always filter solvents, small particles can permanently block the capillaries. Avoid the use of the following steel-corrosive solvents:

- Solutions of alkali halides and their respective acids (for example, lithium iodide, potassium chloride, and so on).
- High concentrations of inorganic acids like nitric acid, sulfuric acid especially at higher temperatures (replace, if your chromatography method allows, by phosphoric acid or phosphate buffer which are less corrosive against stainless steel).
- Halogenated solvents or mixtures which form radicals and/or acids, for example:

  $2CHCl_3 + O_2 \rightarrow 2COCl_2 + 2HCl$

  This reaction, in which stainless steel probably acts as a catalyst, occurs quickly with dried chloroform if the drying process removes the stabilizing alcohol.

- Chromatographic grade ethers, which can contain peroxides (for example, THF, dioxane, di-isopropylether) such ethers should be filtered through dry aluminium oxide which adsorbs the peroxides.
- Solutions of organic acids (acetic acid, formic acid, and so on) in organic solvents. For example, a 1-% solution of acetic acid in methanol will attack steel.
- Solutions containing strong complexing agents (for example, EDTA, ethylene diamine tetra-acetic acid).
- Mixtures of carbon tetrachloride with 2-propanol or THF.

Agilent Technologies on Internet

For the latest information on products and services visit our worldwide web site on the Internet at:

http://www.agilent.com

Select “**Life Sciences & Chemical Analysis Solutions**”

It will provide also the latest firmware of the Agilent 1200 Series modules for download.

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In This Book

This manual contains user information about the Agilent 1200 Series Refractive Index Detector. The manual describes the following:

- introduction to the RI detector and theory of operation,
- site requirements and specifications,
- installing the RI detector,
- using the RI detector,
- optimizing the RI detector,
- troubleshooting overview,
- maintaining and repairing the RI detector.

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